2 f
12 c

GEORGIA INSTITUTE OF TECHNOLOGY
STATE ENGINEERING EXPERIMENT STATION
ATLANTA, GEORGIA

PROGRESS REPORT NO. 1-7
AND
FINAL REPORT

PROJECT NO. 208-156

THE PARTIAL ACETYLATION OF COTTON

J. L. TAYLOR
PROJECT DIRECTOR

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

JUNE 1952-MARCH 1954

CONTENTS

Georgia Institute of Technology
STATE ENGINEERING EXPERIMENT STATION
Atlanta, Georgia

PROGRESS REPORT NO. 1

PROJECT NO. 208-156

THE PARTIAL ACETYLATION OF COTTON

By

J. L. TAYLOR and W. C. CARTER

- o - o - o - o -

CONTRACT NO. A-ls-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

JUNE 20, 1952

Georgia Institute of Technology
STATE ENGINEERING EXPERIMENT STATION
Atlanta, Georgia

PROGRESS REPORT NO. 1

PROJECT NO. 208-156

THE PARTIAL ACETYLATION OF COTTON

By

J. L. TAYLOR and W. C. CARTER

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

JUNE 20, 1952

TABLE OF CONTENTS

This Report Contains 4 Pages

I. SUMMARY

The dyeing equipment to be used in determining the acetylation characteristics of various cottons has been modified, and several trial acetylations have been made in order to determine how well the temperature of acetylation can be controlled and, also, how evenly the cotton is acetylated. The results of these trial runs are not complete.

Twelve lots of cotton varying widely in their properties have been received from the Southern Regional Laboratory, New Orleans, Louisiana. As soon as an acetylation technique has been established, the acetylation characteristics of these cottons will be determined.

II. STATEMENT OF PROBLEM

The purpose of this research program is to determine the relationships between cottons of different properties and their responses to acetylation.

III. SCOPE OF PROBLEM

It has been found that cotton fiber can be chemically modified so as to improve its resistance to microbiological rotting, mildewing and heat degradation. Partially acetylated cotton is an example of such a modification. However, various cottons differ in their response to acetylation, i.e., they have different rates of acetylation. At present, there are no known tests, other than acetylation itself, for determining differences in the extent of acetylation for various cottons. Qualitatively, it has been found that the degree of maturity of cotton fibers has an effect on the acetylation characteristics; however, this is undoubtedly not the entire answer. Other characteristics of cotton in its natural state, as well as in its preparation for acetylation, may influence the reaction

rate. Some of these properties are listed here:

1. cotton variety
2. area of growth
3. fiber maturity
4. fiber fineness
5. fiber strength
6. per cent crystalline cellulose
7. x-ray angle (crystallite orientation)
8. alcohol-soluble content
9. wax content
10. acetic acid-soluble content
11. ash content
12. water-soluble ash constituents
13. water-soluble reducing constituents
14. moisture regain

The problem is to determine whether or not any of these properties can be related quantitatively to the acetylation characteristics. If not, what other properties influence the reaction rates?

IV. PREPARED EXPERIMENTAL METHODS

A. Source of Cottons

Cottons representing a very wide diversity of properties will be furnished by the Southern Regional Research Laboratory, New Orleans, Louisiana.

B. Treatment of Cotton Samples Prior to Acetylation

1. Since these samples may be chosen from various stages of mechanical processing; i.e., bale samples, opener samples, picker lap samples

and sliver samples, it is important that the amounts of extraneous matter normally removed during processing be known. This will be accomplished by processing samples from each lot of cotton through a Shirley Analyzer. Since the results obtained in this program will be ultimately applied to the treatment of yarns and fabrics in which most of the extraneous materials have been removed, it is felt that the Shirley Analyzer should be employed prior to a determination of the acetylation characteristics.

2. In all of the initial studies to find differences in the rates of acetylation for various cottons, the samples will be similarly conditioned, e.g., 65 per cent relative humidity and 70° F.

C. Acetylation Procedure

1. Equipment

All of the acetylations will be carried out in a one-pound Morton package dyeing machine which has been modified so that it is possible to use cotton raw stock instead of yarn packages. The steam jacket which surrounds the dyeing chamber has been modified so that ice water can be circulated around the chamber to maintain the low temperatures necessary in partial acetylation. This machine is constructed so that both open and closed circulation of the reaction mixture is possible. It is estimated that several cotton samples totaling approximately 8 oz. in weight can be treated simultaneously.

2. Presoaking, Acetylation and Analysis of Acetyl Content

In the initial phase of this work, the conditions employed in the presoaking and acetylation, as well as the methods of analysis for

the degree of acetylations, will be those employed at the Southern Regional Research Laboratory.*

V. PRESENT STATUS AND FUTURE PROGRAM

The dyeing machine to be used in these acetylation studies has been modified and some trial runs have been made in order to determine how well the temperature can be controlled during the reaction and how evenly will be the degree of acetylation.. The initial results are encouraging..

Twelve lots of cottons, together with data on some of their physical characteristics, have been received from the Southern Regional Research Laboratory. As soon as it can be established that the acetylation procedure gives controlled, uniform results, the acetylation studies of these twelve samples will be started.

VI. PERSONNEL

These are the personnel currently employed on this project.

Dr, J. L. Taylor	Faculty Research Associate
Dr. W. C. Carter	Faculty Research Associate
Ben G. Holloway	Graduate Student in Textile Engineering
Douglas Wheeler	Senior in Textiles
Demetrios Dellis	Graduate Student in Chemistry

Approved:

[redacted]

Gerald A. Rosselot, Director
State Engineering Experiment Station

Respectfully submitted::

[redacted]

J. L. Taylor,
Project Director

* - - - -
Cooper, A. S., Voorhies, S. T., Jr., Buras, E. M., Jr., and Goldthwart, C. F., "Partial Acetylation of Cotton." Textile Industries 116, No. 1, 97-102, 194-195 (1952).

Georgia Institute of Technology
STATE ENGINEERING EXPERIMENT STATION
Atlanta, Georgia

PROGRESS REPORT NO. 2

PROJECT NO. 208-156

THE PARTIAL ACETYLATION OF COTTON

By

J. L. TAYLOR and W. C. CARTER

- o - o - o - o -

CONTRACT NO. A-ls-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

SEPTEMBER 20, 1952

Georgia Institute of Technology
STATE ENGINEERING EXPERIMENT STATION
Atlanta, Georgia

PROGRESS REPORT NO. 2

PROJECT NO. 208-156

THE PARTIAL ACETYLATION OF COTTON

By

J. L. TAYLOR and W. C. CARTER

- o - o - o - o -

CONTRACT NO. A-ls-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

SEPTEMBER 20, 1952

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

This Report Contains 14 Pages

I. SUMMARY

Techniques for acetylating several samples of cotton simultaneously in a single-package Morton dyeing machine have been worked out so as to maintain close temperature control during acetylation.

The results of seven acetylations, using 12 different cotton samples in each acetylation run, indicate that temperature control is a very important factor in determining the amount of acetylation for a given period of time. An analysis of the results also indicates that considerable acetylation takes place during the first few minutes of the reaction of the acetylation mixture with cotton. Thus, further study should be made of the acetylation of cottons at short periods of acetylation time and at constant temperature, as well as of the effect of temperature on the rate of acetylation at constant time intervals.

II. EXPERIMENTAL WORK

A. Materials

Twelve lots of cotton have been received from the Southern Regional Research Laboratory. The names and characteristics of these cottons are shown in Table I. It is noted that they represent cottons whose characteristics, with respect to fiber maturity and fineness, are considerably different.

B. Sampling

The methods employed in preparing test samples for partial acetylation from bulk laboratory samples were those given in the "Tentative General Methods of Testing Cotton Fibers," A.S.T.M. Designation: D 414-49T.

TABLE I

COTTONS RECEIVED FROM THE SOUTHERN REGIONAL RESEARCH LABORATORY

Cotton	Staple	Maturity		Fineness		Pressley Index	X-Ray Angle	
		NaOH	Arealometer	Weight	Micronaire		40%	50%
1. Indian (J & J)	3/4	93	--	8.2	---	--	34.98	---
2. Sea Island	1-3/4	86	--	---	2.4	8.66	31.36	26.40
3. Memphis	--	38	41	---	1.3	--	37.92	---
4. Empire Bale 92	--	72	72	4.3	3.7	--	34.08	---
5. Stoneville 2B Bale 616654	1-3/32	79	77	---	3.9	7.4	---	---
6. Bob Shaw	--	--	88	---	5.1	--	31.32	---
7. Stoneville 2B Bale 249290	1-1/16	80	--	4.0	3.2	7.7	31.20	---
8. Acala 1517	--	86	84	3.9	4.0	--	29.76	---
9. SXP Bale 3109	1-1/2	86	--	3.2	---	8.3	---	30.03
10. Lockett 140	--	92	--	5.6	5.6	--	36.18	---
11. Pima 32	1-3/8	84	--	---	2.8	10.53	29.28	---
12. Hopi Acala 50	1-1/8	95	--	4.0	4.6	8.6	33.75	---

C. Details of Acetylation Procedure and Analytical Procedure

1. Preparation of Sample

Before starting each acetylation, all the laboratory test samples of 15 g. each were conditioned at approximately 65 per cent relative humidity and 70° F. They were then placed in cotton bags made from open mesh cotton fabric.

2. Presoaking

The conditioned samples were soaked overnight in a closed vessel in glacial acetic acid at room temperature.

3. Preparation of Acetylating Mixture

The acetylating mixture consisted of glacial acetic acid and acetic anhydride in a volume ratio of three to one with 0.15 per cent of 60 per cent by volume perchloric acid, based on the total volume of acetic acid and acetic anhydride. For the one-pound Morton package dyeing machine which was employed in all acetylations, a total volume of approximately 9.62 liters of acetylating mixture was required. This consisted of:

7.2 liters of glacial acetic acid,

2.4 liters of acetic anhydride and

14.2 ml. of 60 per cent perchloric acid.

Two solutions were prepared, one containing 6.2 liters of acetic acid and 2.4 liters of acetic anhydride, and the other containing the remaining liter of acetic acid and the perchloric acid catalyst. Both solutions were precooled to approximately 35° F. before addition to the machine.

4. Acetylation

After several trial runs the following acetylation procedure was employed. The machine was precooled to approximately 65° F. while circulating spent acetylating mixture through the machine. Cooling was accomplished

by circulating ice water in the jacket surrounding the reaction chamber. When the machine and the prepared acetylating mixture had been sufficiently cooled, the machine was drained, and the presoaked samples were rapidly extracted and then placed in the reaction chamber. The machine was closed and the cooled acetic acid-acetic anhydride mixture was added. The solution of catalyst and acetic acid was added in increments of 100 ml. at one-minute intervals until all was added, thus requiring a total of 10 minutes. The acetylating mixture was next circulated for one to two minutes in order to make certain that there were no air pockets, after which a change from open to closed circulation was made. The time at which the machine was closed was considered as time zero in all experimental runs. The acetylating mixture was kept cool by the circulation of ice water in the jacket surrounding the samples. After the completion of an acetylation, the mixture was drained and the samples were thoroughly washed with copious amounts of cold water. The residual amount of acetic acid was neutralized with dilute NH_4OH, followed by a thorough final wash.

5. Analysis of Acetylated Samples

a. Dyeing. In order to determine the evenness of acetylation, portions of the dried acetylated samples were dyed with a mixture of a blue direct color and a yellow acetate color. The dyes employed were Chlorantine Fast Blue 3RLL and Celliton Fast Yellow RRA. The dyeing procedure was the same as that employed at the Southern Regional Research Laboratory.*

---

*Cooper, A. S., Voorhies, S. T., Jr., Buras, E. M., Jr., and Goldthwait, C. F., "Partial Acetylation of Cotton." Textile Industries 116, No. 1, 97-102, 194-195 (1952).

b. Determination of Acetyl Content. Accurately weighed oven-dry samples of approximately 1.5 g. were taken for analysis. Each sample was cut to extremely short fibers in a Wiley Mill.* To each was added 50 ml. of methanol, and each was heated for 15 minutes at 120° F., after which 50 ml. of 0.5 N NaOH was added. The mixture was heated at 140° F. for 30 minutes and was then allowed to stand with occasional agitation for 48 hours before analysis. An alternate method was to heat the samples for one hour at 140° F. before titrating. The mixture was titrated with 0.5 N HCl to the phenolphthalein end point. A blank consisting of a weighed sample of cotton, 50 ml. of alcohol and 50 ml. of 0.5 N NaOH was also titrated with the 0.5 N HCl. The per cent acetyl content was calculated as follows:

$$\% \text{ acetyl content} = \frac{(V_R - V_S)\ (N_A)\ (0.04302)}{W} \times 100$$

where V_B = volume HCl (ml.) required by blank,

V_S = volume HCL (ml.) required by sample,

N_A = normality of HCl,

0.04302 = milliequivalent weight of the acetyl group and

W = dry weight of sample.

III. RESULTS AND DISCUSSION OF RESULTS

The experimental procedure given above was employed in all acetylations made to date. A description of each run is given in Table II. It is noted that the initial and final temperatures are recorded, the initial temperature being the temperature of the acetylating mixture when all of

*The fibers in Run 3 were not cut up before titration.

the catalyst solution had been added and the machine had been changed from open to closed circulation. In all of these runs except Run 3, samples of the 12 cottons listed in Table I were acetylated. In Run 3 some card sliver taken from the yarn-processing department of the Textile School, Georgia Institute of Technology, was used. The procedure for acetylation was identical with that used on S.R.R.L. cotton. The purpose of this run, as well as of several other previous runs not recorded in this report, was to establish operating conditions and to determine the uniformity of acetylation.

TABLE II

TIMES AND TEMPERATURES USED IN ACETYLATING

Run Number	Time of Acetylation (Min.)	Initial Temperature (°F.)	Final Temperature (°F.)
3	80	68	67
4	80	72	71
5	50	68	73
6	50	69	69
7	65	65.5	68
8	65	65	67
9	10	61	65

The addition of one-fifth of the catalyst solution caused the temperature of the acetylating mixture to rise 4° to 6° F., while the addition of the remaining catalyst solution never resulted in more than an additional 1° F. rise.

The acetyl contents of the acetylated samples are shown in Tables III and IV. Table III contains the data obtained from Run 3 in which all of the samples used were obtained from the same source. The wide variation in per cent acetyl content in Table III can be attributed to the fact that these samples were not cut up before saponification. Table IV contains the results for the acetylation of cottons listed in Table I.

The averages of per cent acetyl content for different times of acetylation are shown graphically in Figures 1, 2, 3 and 4. It is noted that the degree of acetylation for 65 minutes is less than that at 50 minutes. This probably is due to the fact that the temperature of this run was slightly low, thus indicating the importance of close temperature control during the time of acetylation. Spot checks in a duplicate run (Run 8) confirm the results obtained in Run 7.

The acetylation results obtained at 80 minutes indicate that the difference in acetylation characteristics of various cottons diminishes when acetylation is carried out at a sufficiently long time. The acetyl contents at 80 minutes vary from 26.58 to 30.65 per cent, or a spread of 4.07 per cent, whereas the spreads in the 50- and 65-minute runs (Runs 5 and 7) are 7.7 and 10.4 per cent, respectively.

Run 9 was made to determine the extent of acetylation during the period that the catalyst was being added to the acetylating bath. It is obvious from these results that considerable acetylation is accomplished during the early stages of acetylation.

Evaluation of all acetylated samples has been limited to the determination of the acetyl content of each acetylated cotton and the dye test for acetylation uniformity. The results of the dye tests indicate that the

TABLE III

VARIATION OF ACETYLATION OF DUPLICATE SAMPLES

Cotton	Sample	Per Cent Acetyl Content
Card sliver from Yarn-Manufacturing Department, Georgia Institute of Technology	1	23.67
	1	23.91
	2	22.20
	2	24.01
	3	24.30
	3	23.58
	4	22.36
	4	23.31
	5	24.26
	5	23.27
	6	22.73
	6	---
	7	23.56
	7	23.37
	8	23.50
	8	24.17
	9	23.63
	9	22.94
Average Per Cent Acetyl Content		23.46 ± 0.47
Standard Deviation		0.602
Standard Error		0.1504

TABLE IV

RESULTS OF THE ACETYLATION OF DIFFERENT COTTONS AT VARIOUS ACETYLATION PERIODS

	Per Cent Acetyl*					
Run	4	5	6	7	8	9
Time (Minutes)	80	50	50	65	65	10
Temperature (°F.)	71-82	68-73	72-69	65-68	65-67	60-65
1. Indian (J & J)	29.33	22.87	22.16	19.98	19.65	8.11
2. Sea Island	30.65	23.66	22.37	21.47	21.14	6.51
3. Memphis	29.86	26.31	23.15	24.96	---	10.51
4. Empire Bale 92	29.05	19.59	19.08	17.90	---	5.76
5. Stoneville 2B Bale 616654	27.81	19.53	18.51	16.02	16.20	5.21
6. Bob Shaw	26.82	16.32	15.44	14.85	---	4.42
7. Stoneville 2B Bale 249290	28.35	19.85	20.83	19.57	---	8.29
8. Acala 1517	30.02	19.32	22.19	21.77	---	8.64
9. SXP Bale 3190	26.58	15.88	16.40	14.29	---	5.51
10. Lockett 140	26.90	20.27	15.73	14.38	13.38	5.82
11. Pima 32	29.05	18.46	20.87	20.69	---	4.96
12. Hopi Acala 50	28.97	19.71	20.84	19.67	18.37	7.31

*The per cent acetyl is the average of two determinations.

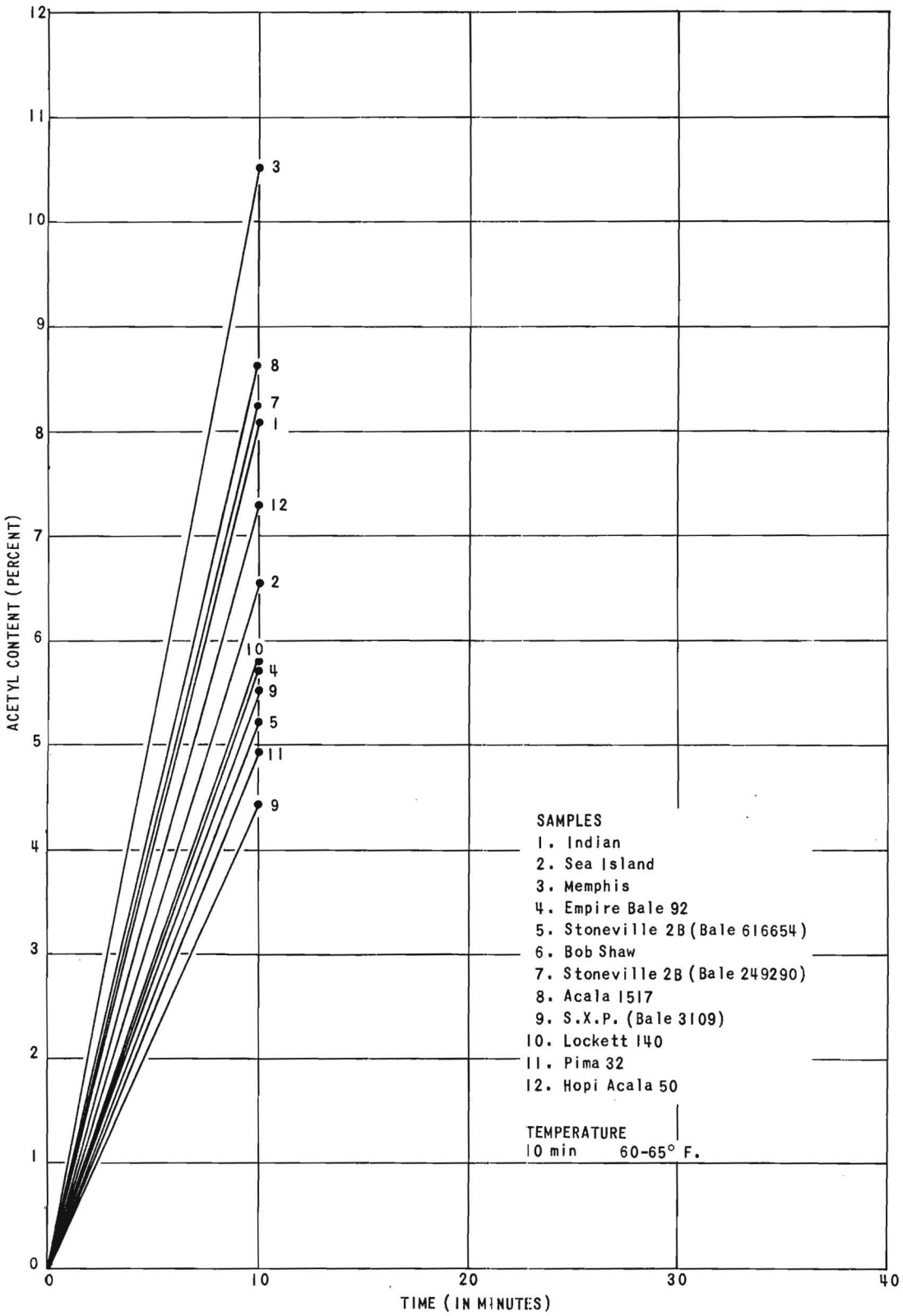

Figure 1. The Change in Acetyl Content with Time of Acetylation.

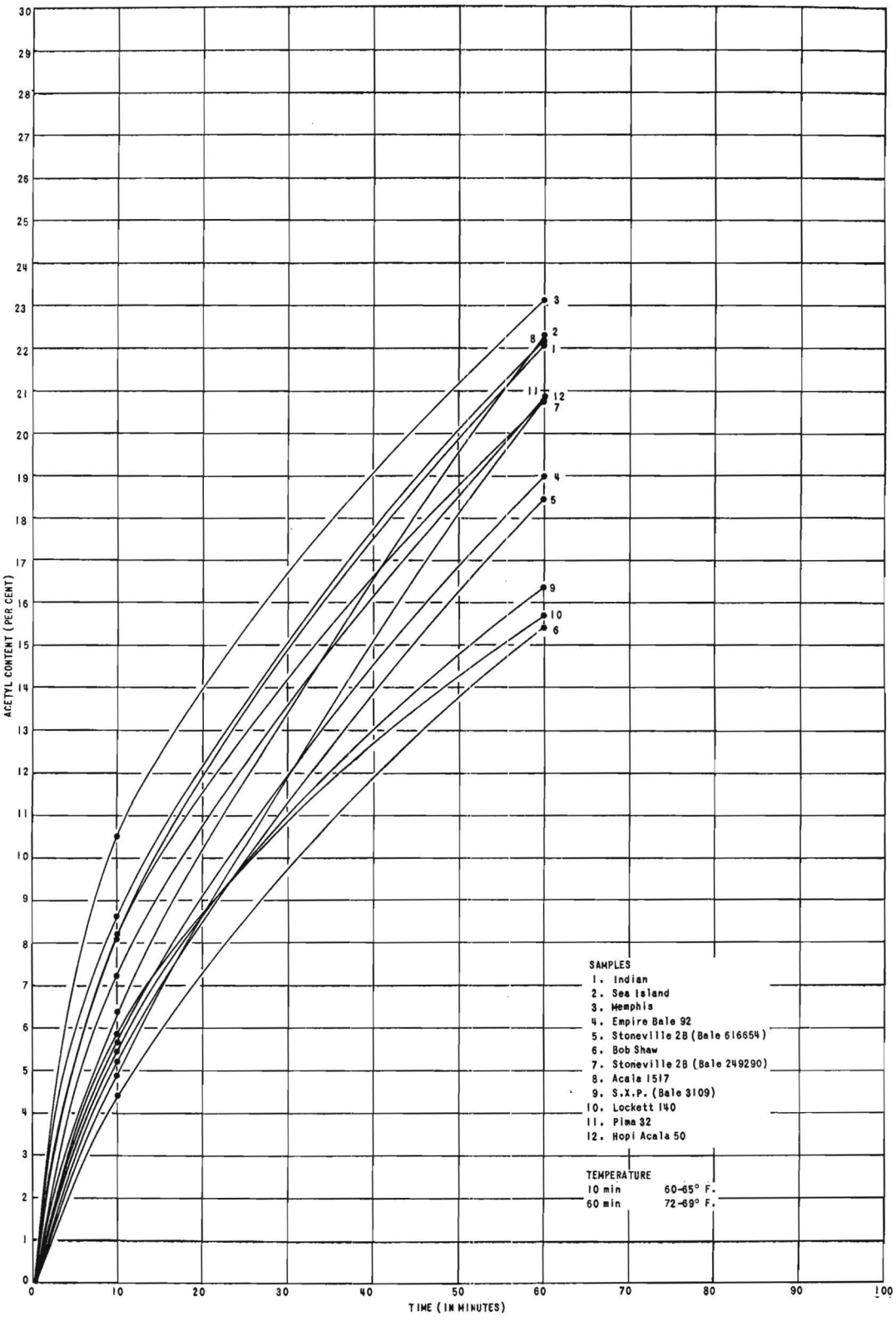

Figure 2. The Change in Acetyl Content with Time of Acetylation.

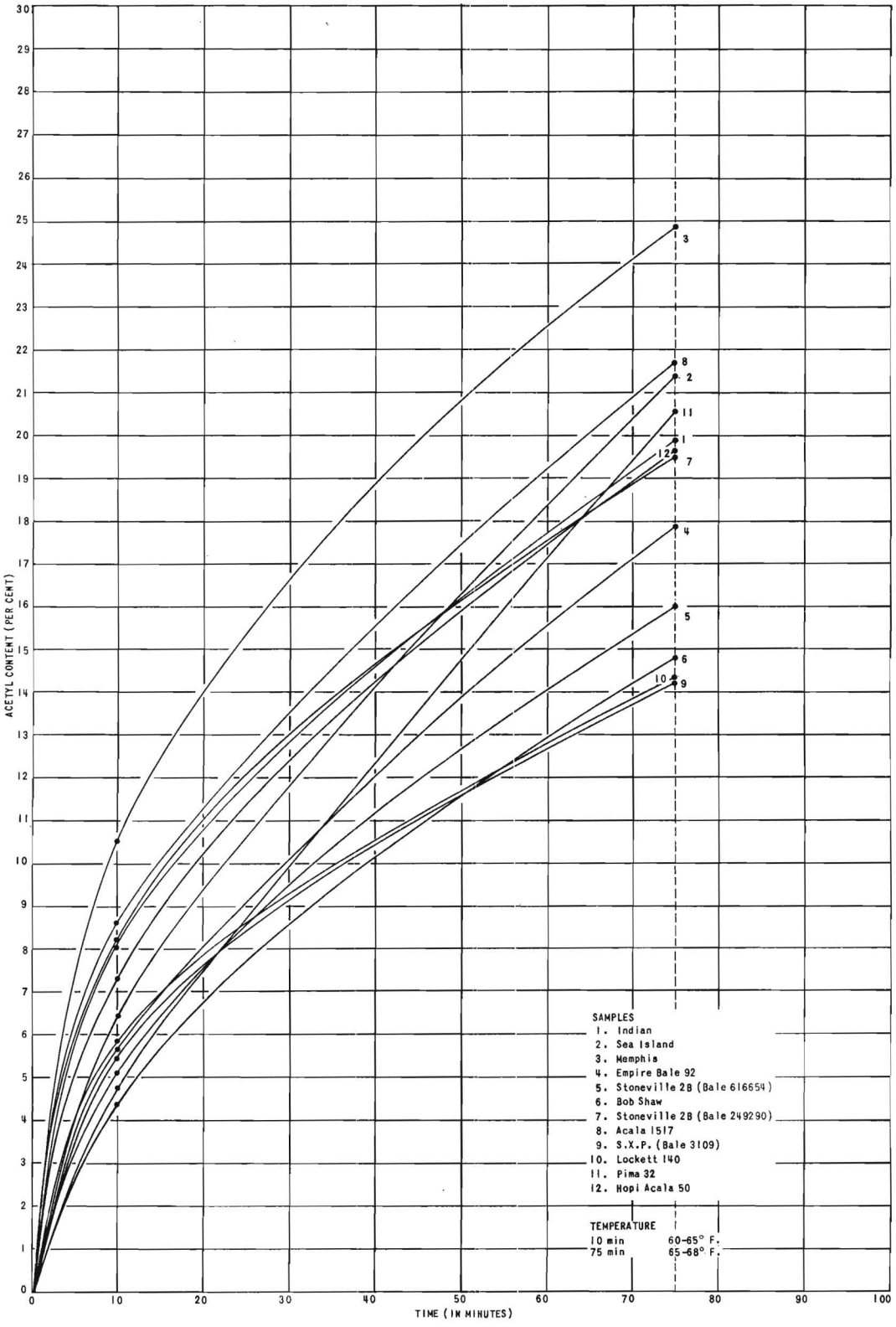

Figure 3. The Change in Acetyl Content with Time of Acetylation.

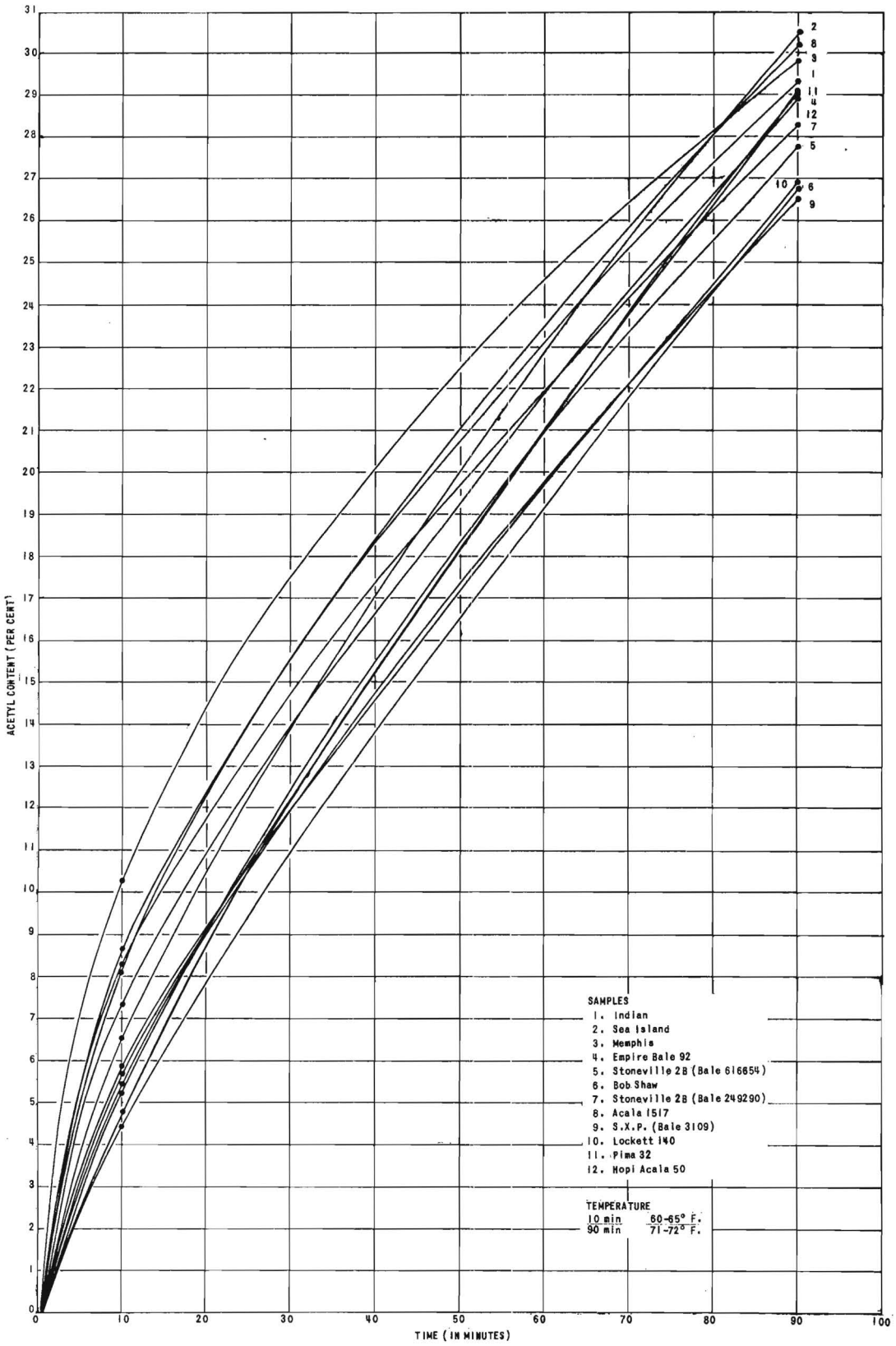

Figure 4. The Change in Acetyl Content with Time of Acetylation.

evenness of acetylation varies with the time of reaction. Thus the 80-minute acetylation gave evenly acetylated samples while the shorter 50- and 10-minute periods contained yellow and blue spots, indicating uneven acetylation.

IV. FUTURE PROGRAM

Plans for future work call for further study of the rate and degree of acetylation of the original twelve samples of cotton supplied by the Southern Regional Research Laboratory, as well as the study of acetylation of additional samples which will also be furnished by S.R.R.L.

It is proposed to use shorter acetylation periods in the range of 5- to 10-minute increments of time. This will give a much closer check on the acetylation rates.

In this study it is also planned to maintain close temperature control, having all acetylation temperatures as nearly the same as possible.

This is to be accomplished by mixing the acetic acid, acetic anhydride and perchloric acid and cooling the mixture to a given temperature before adding it to the cotton to be acetylated.

Respectfully submitted:

J. L. Taylor,
Project Director

Approved:

Gerald A. Rosselot, Director
State Engineering Experiment Station

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 3

PROJECT NO. 208-156

THE PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

DECEMBER 20, 1952

UNITED STATES DEPARTMENT OF AGRICULTURE AGRICULTURAL RESEARCH ADMINISTRATION BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY	REPORT TO SOUTHERN REGIONAL RESEARCH LABORATORY NEW ORLEANS, LOUISIANA

CONTRACT PROJECT REPORT

CONTRACTING ORGANIZATION	PROJECT LEADERS	
Georgia Tech Research Institute	James L. Taylor	
DEPARTMENT	USDA DESIGNATED REPRESENTATIVE	
Research	Charles F. Goldthwait	
DIVISION	CONTRACT NUMBER	INITIATION DATE
Chemical Sciences	A-ls-33460	March 20, 1952
LOCALITY	REPORT NUMBER	PERIOD COVERED
Atlanta, Georgia	Three	Sept. 20 through Dec. 20, 1952

TYPE OF REPORT: PROGRESS (X) PHASE ( ) ANNUAL ( ) TERMINATION ( )

PROJECT TITLE:

The Partial Acetylation of Cotton

ABSTRACT OF PROGRESS

Acetylation of 12 different cottons during this period confirms previous indications that temperature control is very important if small differences in degree and acetylation rates of various cottons are to be measured.

The modification of the acetylating procedure proposed in Progress Report No. 2 failed to give adequate temperature control. In order to achieve even better temperature control during acetylation, a heat exchanger is being added to the Morton package dyeing machine, after which additional acetylation studies will be made on the same 12 cottons and others supplied by The Southern Regional Research Laboratory (S.R.R.L.).

No attempts have been made to correlate the present acetylation data with physical properties of cottons studied.

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 3

PROJECT NO. 208-156

THE PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

DECEMBER 20, 1952

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

This Report Contains 10 Pages.

I. SUMMARY

Acetylation of 12 different cottons during this period confirms previous indications that temperature control is very important if small differences in degree and acetylation rates of various cottons are to be measured.

The modification of the acetylating procedure proposed in Progress Report No. 2 failed to give adequate temperature control. In order to achieve even better temperature control during acetylation, a heat exchanger is being added to the Morton package dyeing machine, after which additional acetylation studies will be made on the same 12 cottons and others supplied by the Southern Regional Research Laboratory (S.R.R.L.).

No attempts have been made to correlate the present acetylation data with physical properties of cottons studied.

II. EXPERIMENTAL WORK

A. Materials

The samples of cottons used during the working period were the same as those listed in Table I, Progress Report No. 2, September 20, 1952.

B. Acetylation Procedures

Several modifications have been made in the procedure described in a previous report.* One modification was in the method of placing the cotton samples in the acetylation chamber. Instead of using cotton mesh bags to keep samples separated during acetylation, the samples are now separated by means of horizontal wire screens. This method of keeping samples separated insures more uniform circulation of the acetylating

*Progress Report No. 2, Project No. 208-156.

mixture through the cotton. This can be demonstrated by noting the color uniformity of the dyed acetylated samples.

As proposed in Report No. 2, the method of adding the catalyst during acetylation has also been modified. In this series of runs (10 through 19) the catalyst, perchloric acid diluted with one liter of glacial acetic acid, was added to the acetylating mixture of acetic anhydride and acetic acid, and the mixture was then cooled to approximately 50° F. before adding the mixture to the cotton to be acetylated.

Although the acetylating mixture was cooled prior to its circulation through the cottons, a rise in temperature was noted almost immediately when circulation was begun. In all runs attempts were made to hold the acetylating temperature in the range between 60° and 70° F. In most instances the temperature was controlled more closely.

III. DISCUSSION OF RESULTS

Four 10-minute acetylations were made on all 12 cottons in which the acetylating temperature was varied slightly in each acetylation. The results shown in Table I indicate that the temperature is an important factor in determining the amount of acetylation during a given time. This is especially true after a temperature of 64° F. is reached.

Two 15-minute runs were made which also indicate that temperature is apparently more critical than time during acetylation. It should be noted that although these acetylations (Runs 14 and 15) were carried out for a longer time, the per cent acetyl in most instances is greater in the 10-minute acetylation made at a higher temperature.

The data shown in Table II are presented graphically in Figures 1, 2, and 3. In each of these acetylations an attempt was made to hold the temperature constant and to vary the time of acetylation. As shown in the table, it was not possible to control the temperature to an exact range in each acetylation.

The data given thus far emphasize the necessity for close temperature control; therefore, steps are being taken to further modify the Morton package dyeing machine to get a better heat exchange in the system during acetylation. This modification consists of adding a set of stainless steel cooling coils in an ice bath. The acetylating mixture will be pumped through the coils for additional cooling during acetylation.

IV. FUTURE PROGRAM

As a preliminary study for future work, a number of acetylations using one type of cotton have been made in flasks in the laboratory

TABLE I

RESULTS OF THE ACETYLATION OF DIFFERENT COTTONS AT VARIOUS ACETYLATION TEMPERATURES

Run		Per Cent Acetyl*					
		11	12	13	10	14	15
Time (Minutes)		10	10	10	10	15	15
Temperature (°F.)		60-62	62-63	64-66	70-75	64-70	65-73
Average Temp. (°F.)		61.5	63	65	75	67.3	70
1.	Indian (J & J)	3.70	3.89	5.42	12.63	7.68	7.86
2.	Sea Island	3.28	2.95	3.94	11.15	5.05	6.08
3.	Memphis	5.09	5.48	7.84	14.16	8.47	9.70
4.	Empire Bale 92	3.74	2.77	3.81	13.12	4.34	5.56
5.	Stoneville 2B Bale 616654	2.67	2.89	3.40	7.94	3.85	4.45
6.	Bob Shaw	2.18	2.45	2.49	5.04	3.44	4.55
7.	Stoneville 2B Bale 249290	3.38	4.04	4.44	8.81	6.08	7.69
8.	Acala 1517	3.44	3.87	4.05	8.85	6.01	8.89
9.	SXP Bale 3190	2.78	3.32	2.72	7.16	3.20	4.99
10.	Lockett 140	2.21	2.79	1.97	4.75	3.08	5.32
11.	Pima 32	2.85	3.41	2.80	6.93	5.56	8.06
12.	Hopi Acala 50	2.63	3.51	2.51	7.90	5.06	9.04

*The per cent acetyl is the average of two determinations.

TABLE II

RESULTS OF THE ACETYLATION OF DIFFERENT COTTONS AT VARIOUS ACETYLATION PERIODS

Run		Per Cent Acetyl*					
		13	14	16	17	18	19
Temperature (°F.)		64-66	64-70	65-68	65-72	66-69	68-70
Average Temp. (°F.)		65	67.3	68	70	68.5	69.6
Time (Minutes)		10	15	25	35	45	60
1.	Indian (J & J)	5.42	7.68	9.32	12.03	16.16	17.75
2.	Sea Island	3.94	5.05	8.33	11.10	16.18	15.48
3.	Memphis	7.84	8.47	13.73	14.31	19.29	20.08
4.	Empire Bale 92	3.81	4.34	8.01	10.84	12.85	14.21
5.	Stoneville 2B Bale 616654	3.40	3.84	7.04	8.76	11.20	12.60
6.	Bob Shaw	2.49	3.44	6.07	7.74	10.15	11.55
7.	Stoneville 2B Bale 249290	4.44	6.08	9.73	11.61	14.90	16.10
8.	Acala 1517	4.05	6.01	10.67	12.51	15.09	17.24
9.	SXP Bale 3190	2.72	3.20	6.95	8.34	11.02	11.55
10.	Lockett 140	1.97	3.08	7.02	7.68	10.65	10.63
11.	Pima 32	1.93	5.59	9.87	11.24	15.91	16.02
12.	Hopi Acala 50	2.51	5.06	12.28	14.09	16.09	15.16

*The per cent acetyl is the average of two determinations.

in order to show the effects of the acetylating temperature, the catalyst, and the presoaking temperature. The results of these tests are shown in Tables III, IV, and V.

TABLE III

RESULTS OF THE ACETYLATION OF A SINGLE COTTON AT VARIOUS ACETYLATION TEMPERATURES

Acetylating Time--10 Minutes				
Temperature (°F.)	60	65	70	75
Per Cent Acetyl	2.95	4.98	4.40	5.10

TABLE IV

RESULTS OF THE ACETYLATION OF A SINGLE COTTON WITH VARIOUS AMOUNTS OF CATALYST

Acetylating Time--10 Minutes Acetylating Temperature--70° F.				
Drops of Catalyst	2	3*	5	7
Per Cent Acetyl	3.0	3.6	5.2	6.4

*3 drops is equal to 1.15 per cent, the standard amount used in all acetylations

TABLE V

RESULTS OF THE ACETYLATION OF A SINGLE COTTON WITH VARIOUS PRESOAK TIMES

Acetylating Time--10 Minutes Acetylating Temperature--70° F.			
Presoak Time (Minutes at 140° F.)	15	25	45
Per Cent Acetyl	4.35	5.5	5.7

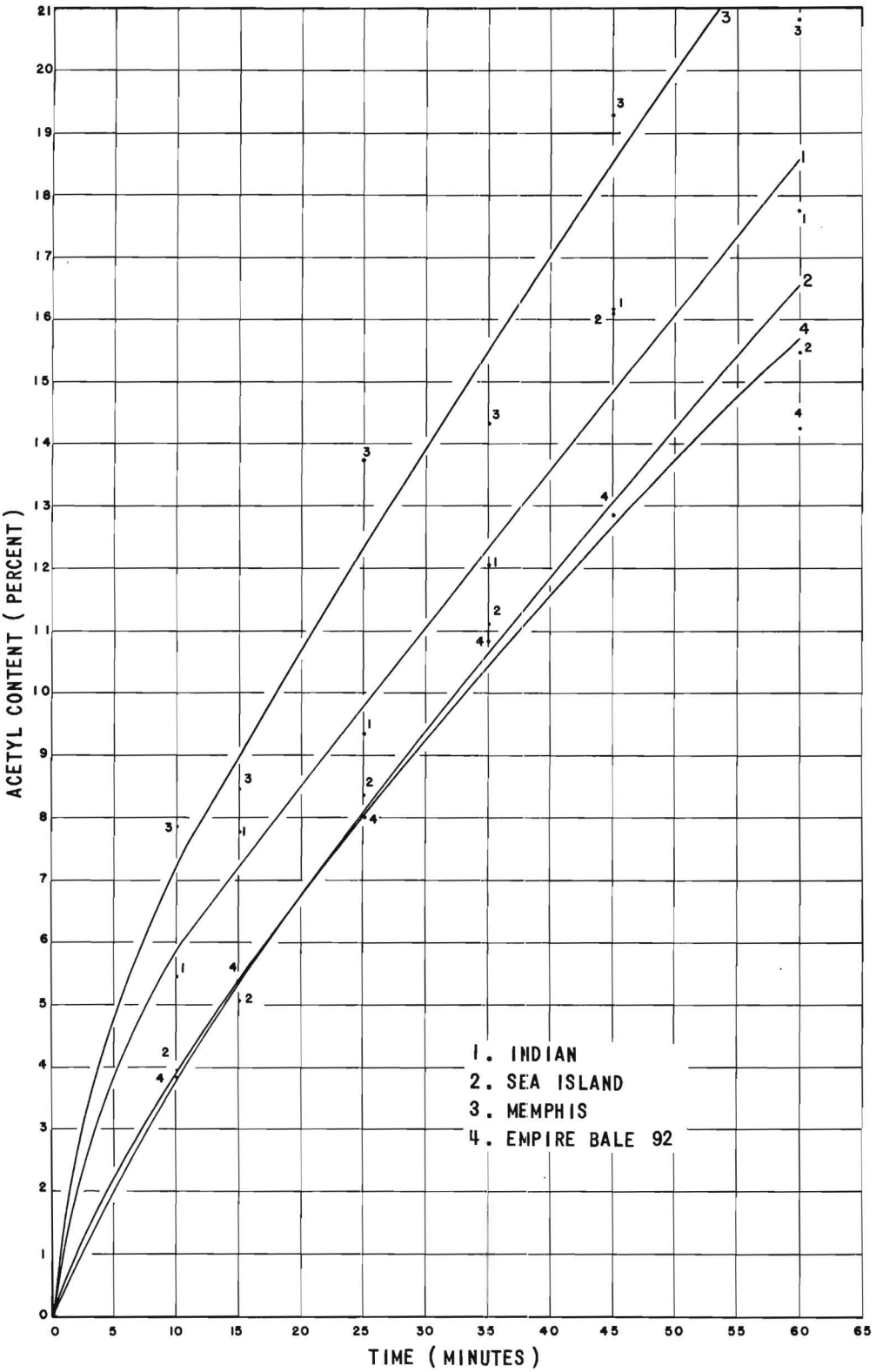

Figure 1. The Change in Acetyl Content With Time of Acetylation.

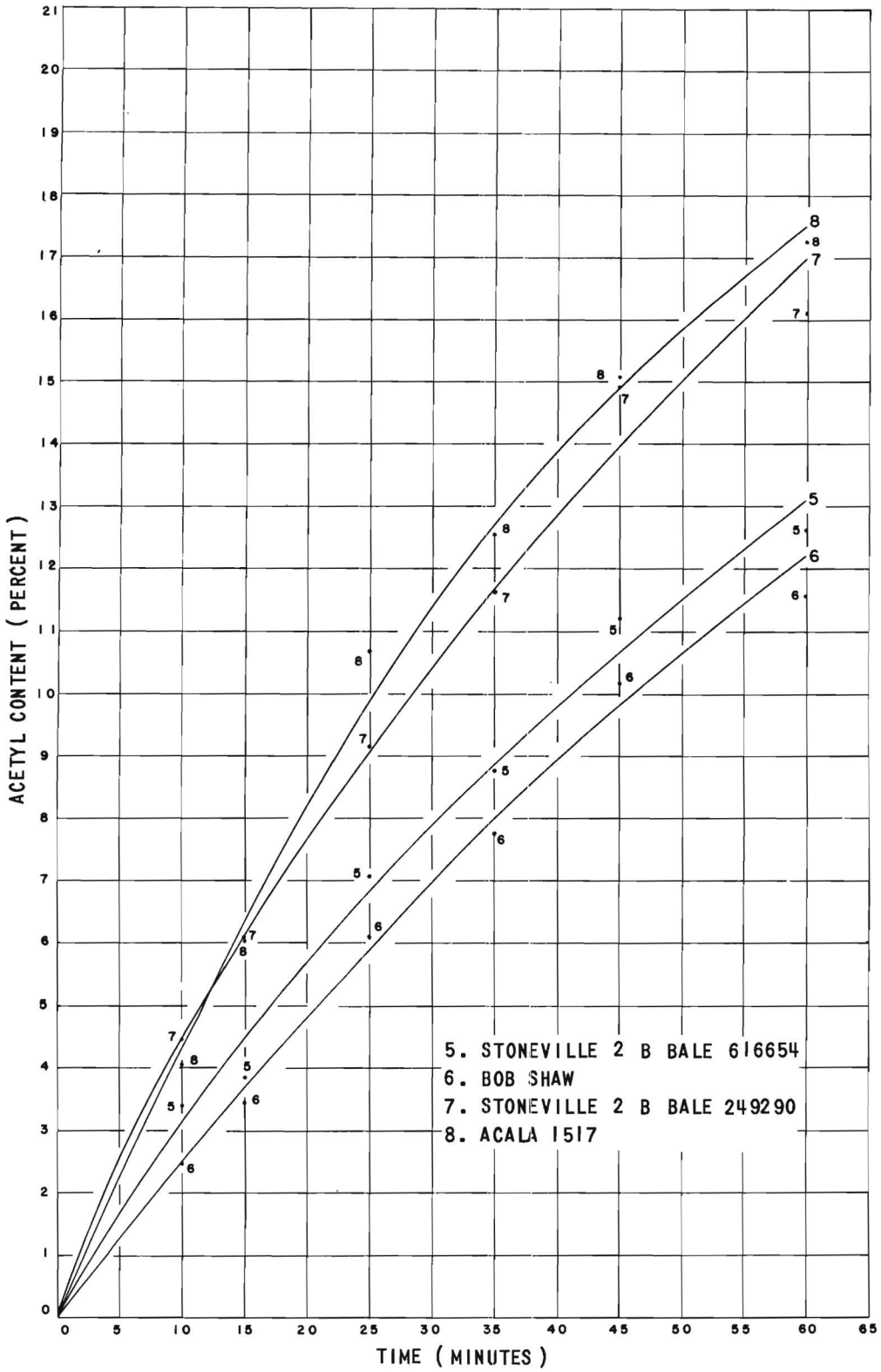

Figure 2. The Change in Acetyl Content With Time of Acetylation.

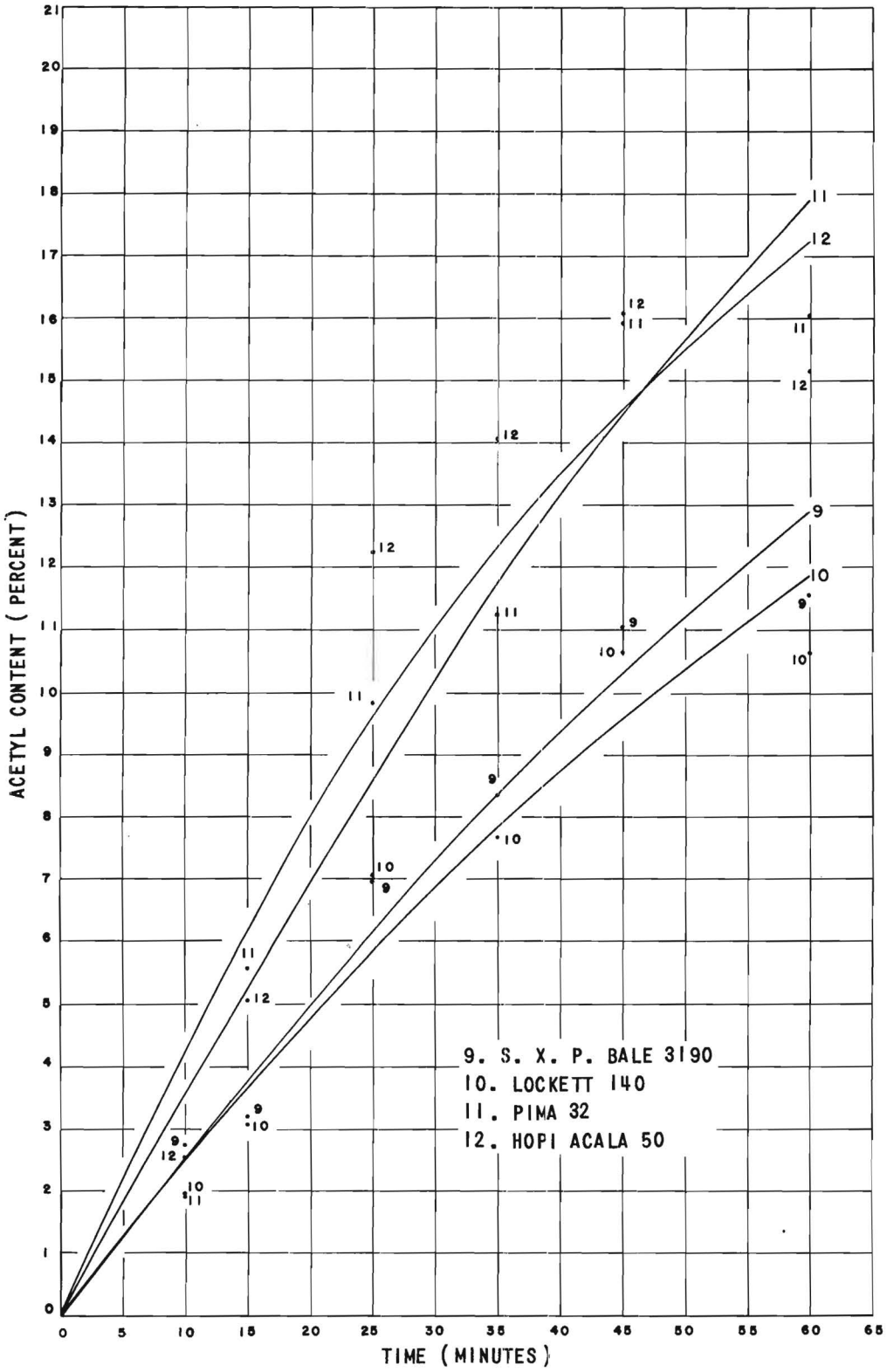

Figure 3. The Change in Acetyl Content With Time of Acetylation.

Additional acetylations will be conducted with the cotton samples on hand, as well as with those being prepared by the S.R.R.L., as soon as the acetylating equipment can be modified to take care of the additional cooling.

Plans are also being formulated to study the effect of moisture content and scouring treatments on the rate and degree of acetylation of certain cottons.

V. PERSONNEL

The following are the personnel currently employed on this project:

Dr. J. L. Taylor	Project Director	
Mr. Alton R. Colcord	Chemical Engineer	Full Time
Mr. Demetrios Dellis	Graduate Student, Chemistry	Part Time
Mr. Walter M. Ligon	Graduate Student, Chemical Engineering	Part Time
Mr. George Adams	Senior, Chemical Engineering	Part Time

Respectfully submitted:

James L. Taylor,
Project Director

Approved:

Herschel H. Cudd, Acting Director
Engineering Experiment Station

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 4

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

MARCH 20, 1953

UNITED STATES DEPARTMENT OF AGRICULTURE AGRICULTURAL RESEARCH ADMINISTRATION BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY	REPORT TO SOUTHERN REGIONAL RESEARCH LABORATORY NEW ORLEANS, LOUISIANA

CONTRACT PROJECT REPORT

CONTRACTING ORGANIZATION	PROJECT LEADERS	
Georgia Tech Research Institute	James L. Taylor	
DEPARTMENT	USDA DESIGNATED REPRESENTATIVE	
Research	Charles F. Goldthwait	
DIVISION	CONTRACT NUMBER	INITIATION DATE
Chemical Sciences	A-1s-33460	March 20, 1952
LOCALITY	REPORT NUMBER	PERIOD COVERED
Atlanta, Georgia	Four	Dec. 20, 1952 - March 20, 1953

TYPE OF REPORT: PROGRESS (X) PHASE ( ) ANNUAL ( ) TERMINATION ( )

PROJECT TITLE:

The Partial Acetylation of Cotton

ABSTRACT OF PROGRESS

During this period considerable progress has been made in the acetylation studies. Although considerable expense and effort was expended in modifying the acetylating equipment, the present results of maintaining a positive temperature control during acetylation justifies the efforts required to make the changes.

Investigations have been conducted on six cottons of widely different physical properties. These investigations included a comparison of the acetylation rates of scoured and unscoured cottons. The results of these studies indicate that scouring tends to minimize the differences in acetyl content of different cottons: unscoured immature cottons acetylate more readily than mature cottons; the effects of maturity and fiber fineness on acetylation diminish after scouring, regardless of the nature of the scouring method. Studies made to determine the effects of moisture content, time and temperature of presoaking prior to acetylation have been undertaken and data presented.

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 4

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

MARCH 20, 1953

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

I. WORK PROGRAM

The work program followed for the past quarter is presented below.

1. Previous work had shown the necessity of close control of temperature during acetylation. After completion of modifications of the machine, acetylation can now be controlled at 64° F ± 0.5°.F.

2. Six varieties of cotton were acetylated at 64° F for 30, 60 and 90 minutes after four different scouring treatments, and the resultant per cent acetyl content was compared with unscoured samples. The results are presented in Table II and shown in Figures 1-11. Scouring greatly increases the rate of acetylation during the first 30 minutes and minimizes the differences in acetylation due to the variety of cotton. Variations in scouring methods had relatively little effect on the acetylation rate.

3. An investigation of the effects of moisture content on the rate and degree of acetylation of six varieties of cotton was undertaken. Acetylation of cotton having moisture content corresponding to 15 per cent R. H. and 65 per cent R. H. have been made. Other acetylations using the same cottons having moisture contents corresponding to 35, 50 and 85 per cent R. H. will be made in order to complete the investigation.

4. Additional studies on the effect of temperature and time of presoaking with glacial acetic acid have been conducted. To date, acetylations have been made on six varieties of cotton

which have been presoaked at 10, 30, 60, 120 and 240 minutes at 70° F, 100° F and 130° F. Analyses have been made on the 100° F series.

Work described in paragraphs 1 and 2 has been completed while work described in paragraphs 3 and 4 is continuing.

II. EXPERIMENTAL WORK

A. Equipment Modification

The results of previous acetylations indicated that it was very important that accurate control of temperature be maintained during acetylations if comparable results were to be obtained when studying acetylation rates.

Thus, in order to maintain a better control over the temperatures during acetylation of cottons, modifications in the Morton dyeing machine were undertaken. These modifications consisted of incorporating a single-pass stainless steel cooling coil into the circulating system of the Morton machine. The cooling coil was mounted in a galvanized tank which can be filled with ice for cooling during acetylation.

Approximately one month was required to draw plans, to obtain the stainless steel material, to fabricate the coils and to make other necessary changes in the valves and fittings on the acetylating machine.

At present, by careful control of the flow of the acetylating mixture through the cooling coil or by control of heating valves, a constant temperature can be maintained at 64° F ± 0.5° F.

B. Acetylation

After the modifications of the cooling and heating system of the acetylating machine, tests were made to ascertain that positive temperature control had been achieved as well as proper acetylation procedures established. These tests showed conclusively that the acetylating temperature can be maintained at a given temperature for any period of time.

Due to the increased volume of the cooling system, increased amounts of acetylating mixture were required to operate the machine. The amounts of materials now used in each acetylation are:

12.6 liters of glacial acetic acid,

4.2 liters of acetic anhydride, and

25.5 ml. of 60 per cent perchloric acid.

The capacity of the acetylating chamber is unchanged, and approximately 240 grams of cotton are acetylated in each run.

The preparation of the acetylating mixture is the same, i. e., the acetic acid and acetic anhydride mixture is cooled to 50° F before adding the required amount of perchloric acid diluted with one liter of glacial acetic acid. The mixture of acetic acid, acetic anhydride and catalyst is again cooled to 50° F before entering it in the acetylating machine.

All acetylations in this series have been maintained at a constant temperature of 64° F.

C. Materials

To conduct the program outlined in Chapter I of this report, six cottons with a wide range of physical characteristics were selected by mutual agreement with the S.R.R.L. These cottons and their physical properties are presented in Table I.

TABLE I

COTTONS SELECTED FOR SCOURING, MOISTURE AND PRESOAKING STUDIES

Cottons	NaOH* Maturity (per cent)	Micronaire Reading	Pressley Index	X-Ray Angle 40%*
Memphis	38	2.45	7.15	37.92
Empire Bale 92	72	3.70	7.30	34.08
Bob Shaw	88**	5.10	8.17	31.32
Stoneville 2B Bale 249290	80	3.20	8.07	31.20
Acala 1517	86	4.00	8.93	29.76
Lockett 140	92	5.67	7.51	36.18

*Data furnished by the Southern Regional Research Laboratory.
**Maturity determined by Arealometer.

It should be noted that a wide range in maturity values, fiber fineness and other properties are demonstrated in the fibers selected.

D. Scouring

The six cottons selected were first scoured, dried and allowed to condition at 65 per cent R. H. at 70° F prior to presoaking and acetylation. Four progressively severe methods of scouring were used on all cottons.

Method I

1.0 per cent Duponal RA
1.5 per cent Tetrasodium pyrophosphate
Scour cotton 30 minutes at the boil.
Rinse in warm water and dry.

Method II

2.0 per cent NaOH
1.0 per cent Merpol C
0.5 per cent Duponal RA
0.5 per cent Tetrasodium pyrophosphate
Scour cotton 60 minutes at the boil.
Rinse in warm water and dry.

Method III

3.0 per cent NaOH
0.1 per cent Triton X-100
0.3 per cent TSPP
Scour cotton 60 minutes at the boil.
Rinse and dry.

Method IV

5.0 per cent NaOH
2. per cent Soap (Neutral Oleate)
Scour cotton 90 minutes at the boil.
Rinse and dry.

All percentages are based on the weight of the cotton, and all scourings were made in a 20:1 bath.

Moisture and micronaire determinations were made on all conditioned (65 per cent R. H. and 70° F) unscoured, scoured and acetylated samples. Results of these determinations are presented in Table II.

After conditioning, the scoured cottons were presoaked in glacial acetic acid for 18 hours at 70° F. Acetylations of 30-, 60-, and 90-minute durations at 64° F were made on each presoaked sample of cotton. The results of the acetyl analyses are also given in Table II and Figures 1-11.

E. Conditioning at Different Relative Humidities

The conditioning of all cotton samples was achieved by placing the cottons in a desiccator containing a sulfuric acid solution whose concentration had been adjusted to give the required relative humidity at 70° F. Tests were made to determine the moisture equilibrium time of the cottons under investigation.

After moisture equilibrium was reached, the samples were immediately transferred to a container of glacial acetic acid and presoaked in the usual manner for 18 hours; all preconditioned samples were acetylated for 30 minutes at 64° F.

Analyses of these acetylations are not complete and will be included in the next progress report.

F. Presoaking at Elevated Temperatures

To study the effect of temperature and time of presoaking cottons in glacial acetic acid, the six varieties of cottons listed in Table I have been presoaked at 70° F, 100° F and 130° F for 10-, 30-, 60-, 120- and 240-minute periods. After presoaking, the acetylation was carried out for 45 minutes at 64° F. Acetyl analyses have been made only on the acetylated samples which were presoaked at 100° F. The results of these

TABLE II

ANALYTICAL DATA FOR ACETYLATED COTTONS AT CONSTANT TEMPERATURE AND AT VARIOUS ACETYLATION TIMES

Cottons	Unacetylated		Acetylated at 64° F								
			30 Minutes			60 Minutes			90 Minutes		
	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire
UNSCOURED:											
Memphis	5.8	2.45	14.10	4.6	2.67	20.0	4.1	2.80	23.7	4.7	3.02
Empire Bale 92	5.8	3.70	8.60	5.1	3.90	14.1	4.4	4.02	19.4	4.7	4.30
Bob Shaw	5.4	5.10	6.55	5.3	5.38	11.1	5.0	5.42	16.3	4.9	5.52
Stoneville 2B Bale 249290	5.9	3.20	8.95	5.0	3.80	15.9	4.5	3.85	20.4	4.4	4.10
Acala 1517	6.0	4.00	9.20	5.2	4.20	14.0	4.9	4.33	20.9	4.8	4.55
Lockett 140	5.9	5.67	6.45	5.4	5.95	10.9	4.9	5.95	14.1	5.2	6.22
SCOUR I:											
Memphis	4.8	2.50	18.3	2.5	2.70	20.80	3.0	2.91	26.1	2.3	3.13
Empire Bale 92	4.6	3.71	16.3	2.5	4.03	20.90	3.0	4.32	24.2	1.7	4.40
Bob Shaw	4.9	5.07	16.1	2.6	5.13	19.70	2.8	5.67	23.8	1.7	5.80
Stoneville 2B Bale 249290	4.8	3.50	16.2	2.6	3.73	20.40	2.8	4.15	23.6	1.8	4.18
Acala 1517	4.8	3.95	15.9	2.6	4.10	21.10	3.0	4.65	25.0	2.0	4.63
Lockett 140	4.7	5.65	15.7	2.6	5.67	19.80	2.8	6.18	24.4	2.0	6.20

(Continued)

TABLE II (Continued)

ANALYTICAL DATA FOR ACETYLATED COTTONS AT CONSTANT TEMPERATURE AND AT VARIOUS ACETYLATION TIMES

Cottons	Unacetylated		Acetylated at 64° F								
			30 Minutes			60 Minutes			90 Minutes		
	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire
SCOUR II:											
Memphis	4.9	2.50	17.9	2.2	2.62	21.20	2.7	2.73	26.1	1.9	2.98
Empire Bale 92	4.6	3.65	16.4	2.5	3.77	20.60	2.6	4.13	25.3	1.6	4.20
Bob Shaw	4.4	5.00	14.9	2.2	5.02	20.00	3.0	5.55	24.8	1.7	5.50
Stoneville 2B Bale 249290	4.8	3.53	16.3	2.3	3.63	20.20	2.7	4.00	25.0	1.8	4.03
Acala 1517	4.6	3.93	17.9	2.2	4.00	21.50	2.7	4.45	30.7	2.1	4.57
Lockett 140	4.4	5.57	15.8	2.1	5.65	20.40	2.6	6.20	25.6	1.7	6.07
SCOUR III:											
Memphis	5.6	2.50	19.2	3.7	2.68	23.2	3.8	2.75	24.6	2.2	2.95
Empire Bale 92	5.0	3.72	17.0	3.6	3.98	22.0	3.4	4.10	23.7	2.6	4.40
Bob Shaw	4.8	5.03	16.0	3.3	5.35	21.5	3.5	5.52	23.0	2.7	5.75
Stoneville 2B Bale 249290	5.1	3.50	16.8	3.4	3.80	22.5	3.6	4.05	23.8	2.7	4.15
Acala 1517	5.4	4.00	17.0	3.5	4.28	22.7	3.8	4.50	24.6	2.7	4.60
Lockett 140	4.8	5.70	15.4	3.2	5.97	20.8	3.4	6.10	23.8	2.6	6.10

(Continued)

TABLE II (Continued)

ANALYTICAL DATA FOR ACETYLATED COTTONS AT CONSTANT TEMPERATURE AND AT VARIOUS ACETYLATION TIMES

Cottons	Unacetylated		Acetylated at 64° F								
			30 Minutes			60 Minutes			90 Minutes		
	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire
SCOUR IV:											
Memphis	5.2	2.53	18.3	4.2	2.63	23.7	3.6	2.92	25.4	2.6	2.95
Empire Bale 92	5.0	3.70	16.7	4.0	4.00	22.9	3.5	4.02	24.5	2.8	4.20
Bob Shaw	5.0	5.00	16.3	3.9	5.32	21.5	3.3	5.52	24.2	2.2	5.53
Stoneville 2B Bale 249290	5.0	3.50	17.6	3.8	3.7	22.5	3.6	3.95	24.8	2.3	4.05
Acala 1517	5.0	3.95	18.0	4.0	4.18	23.3	3.5	4.47	26.0	2.3	4.40
Lockett 140	4.7	5.68	16.3	3.8	6.00	21.9	3.4	6.07	24.8	2.3	6.15

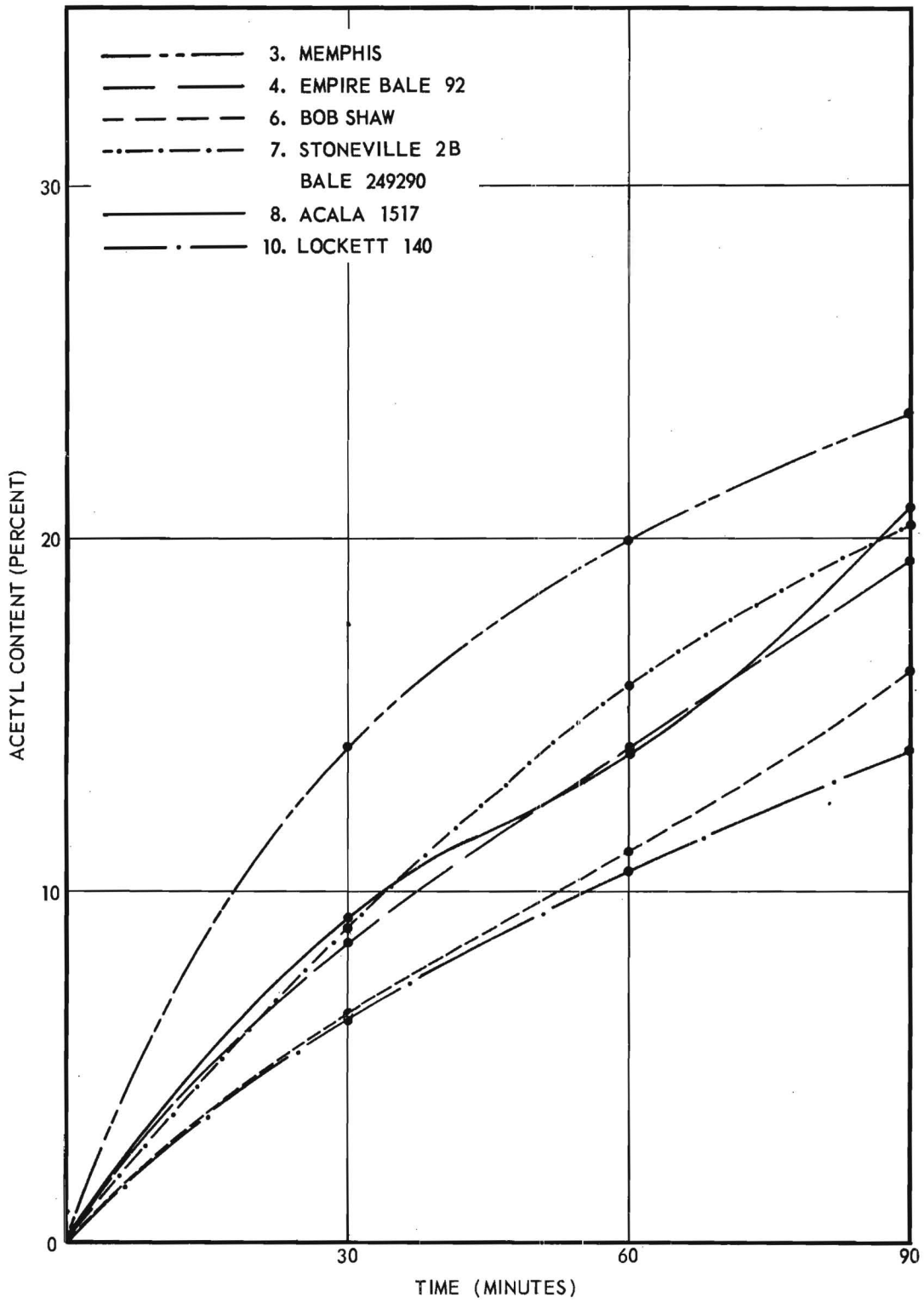

Figure 1. The Change in Acetyl Content With Time of Acetylation for Unscoured Cottons.

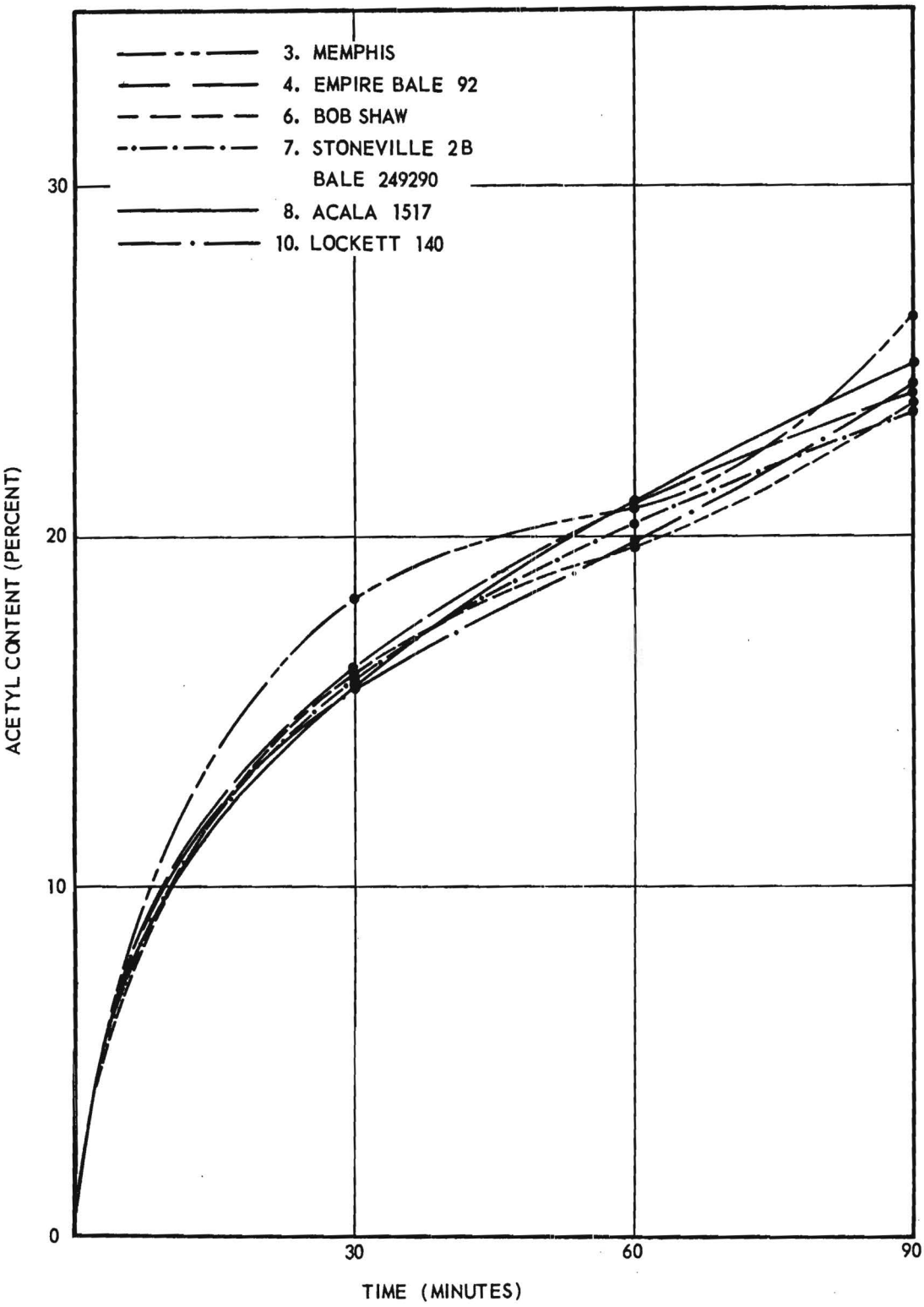

Figure 2. The Change in Acetyl Content With Time of Acetylation for Cottons Scoured by Method I.

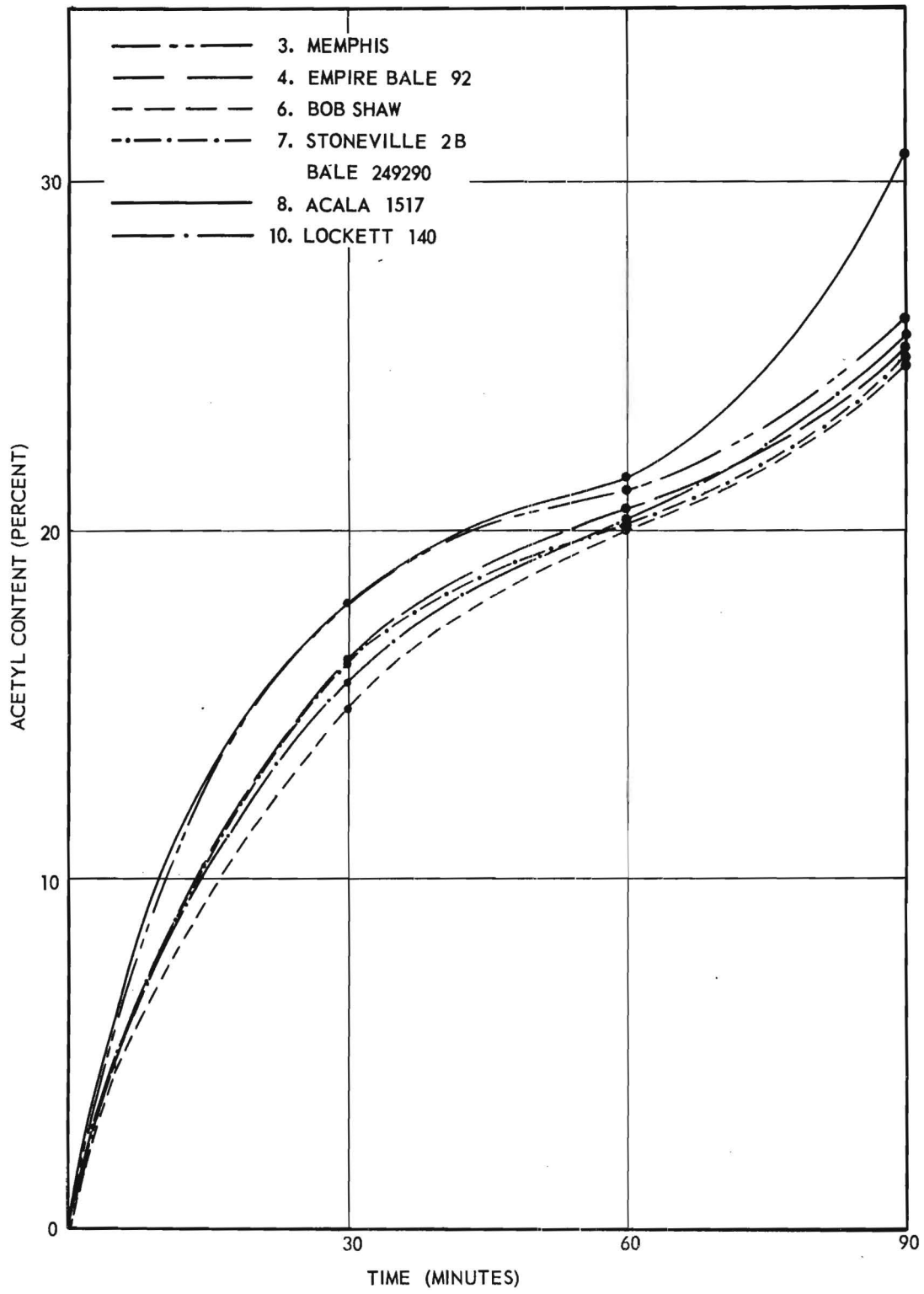

Figure 3. The Change in Acetyl Content With Time of Acetylation for Cottons Scoured by Method II.

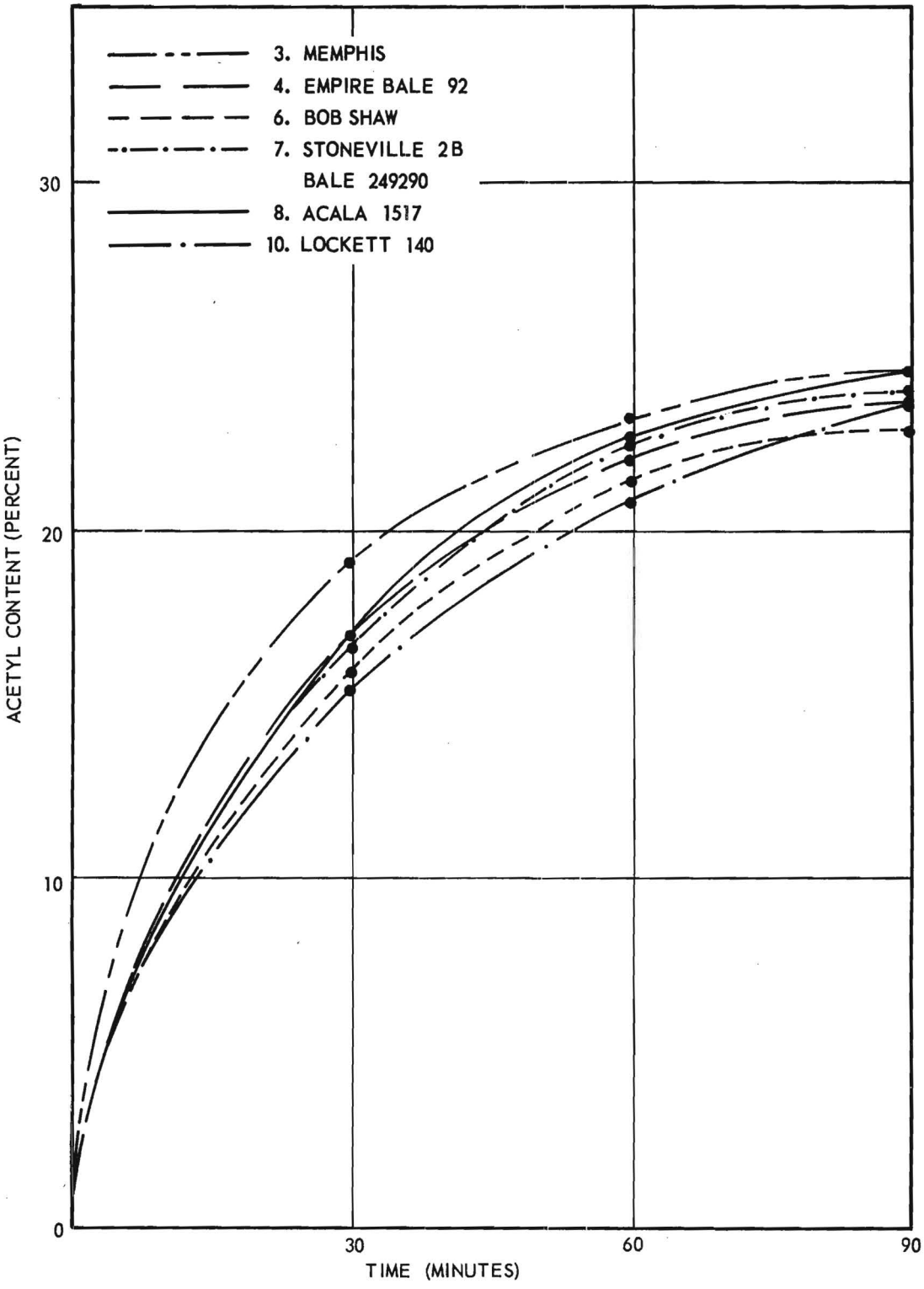

Figure 4. The Change in Acetyl Content With Time of Acetylation for Cottons Scoured by Method III.

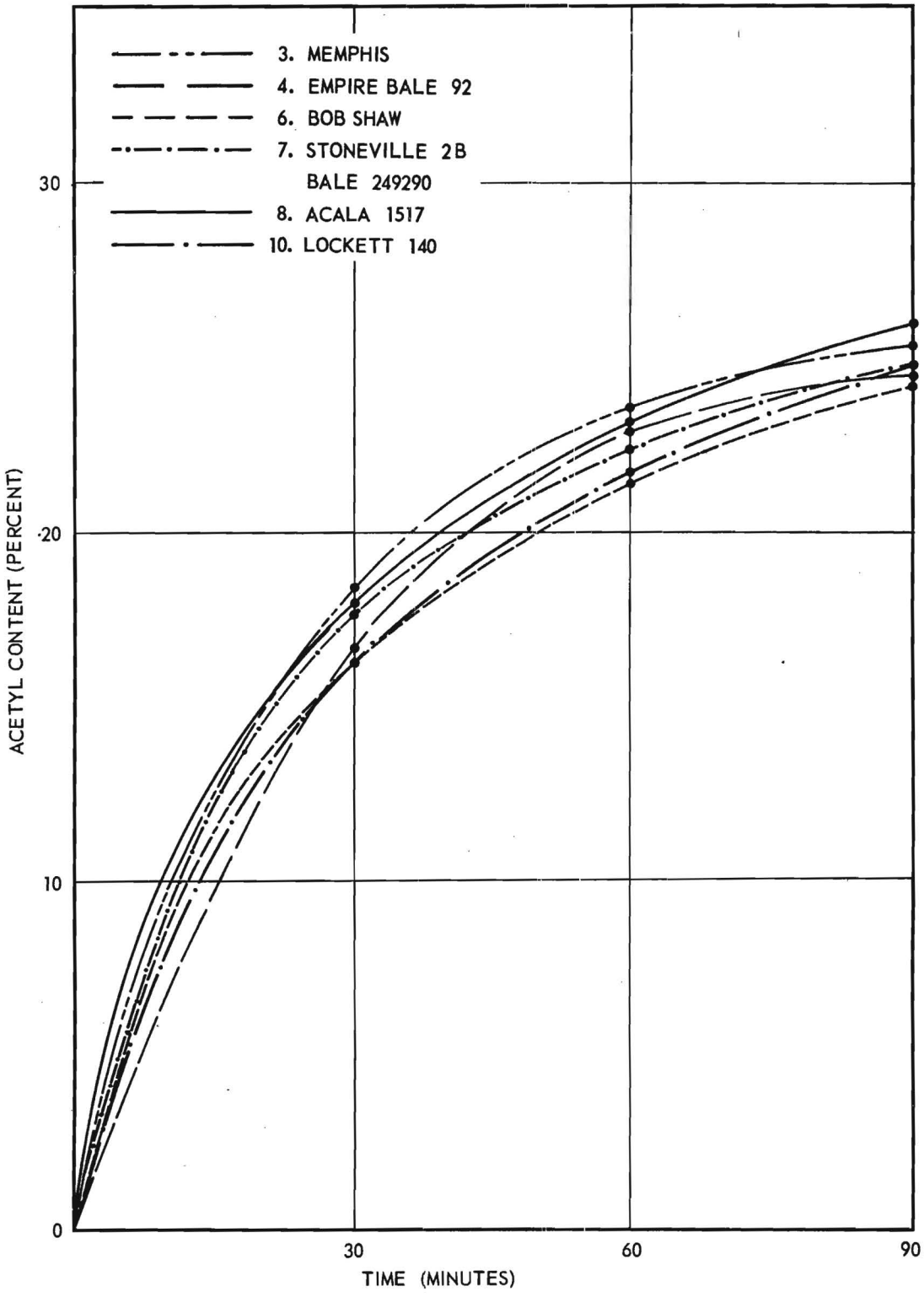

Figure 5. The Change in Acetyl Content With Time of Acetylation for Cottons Scoured by Method IV.

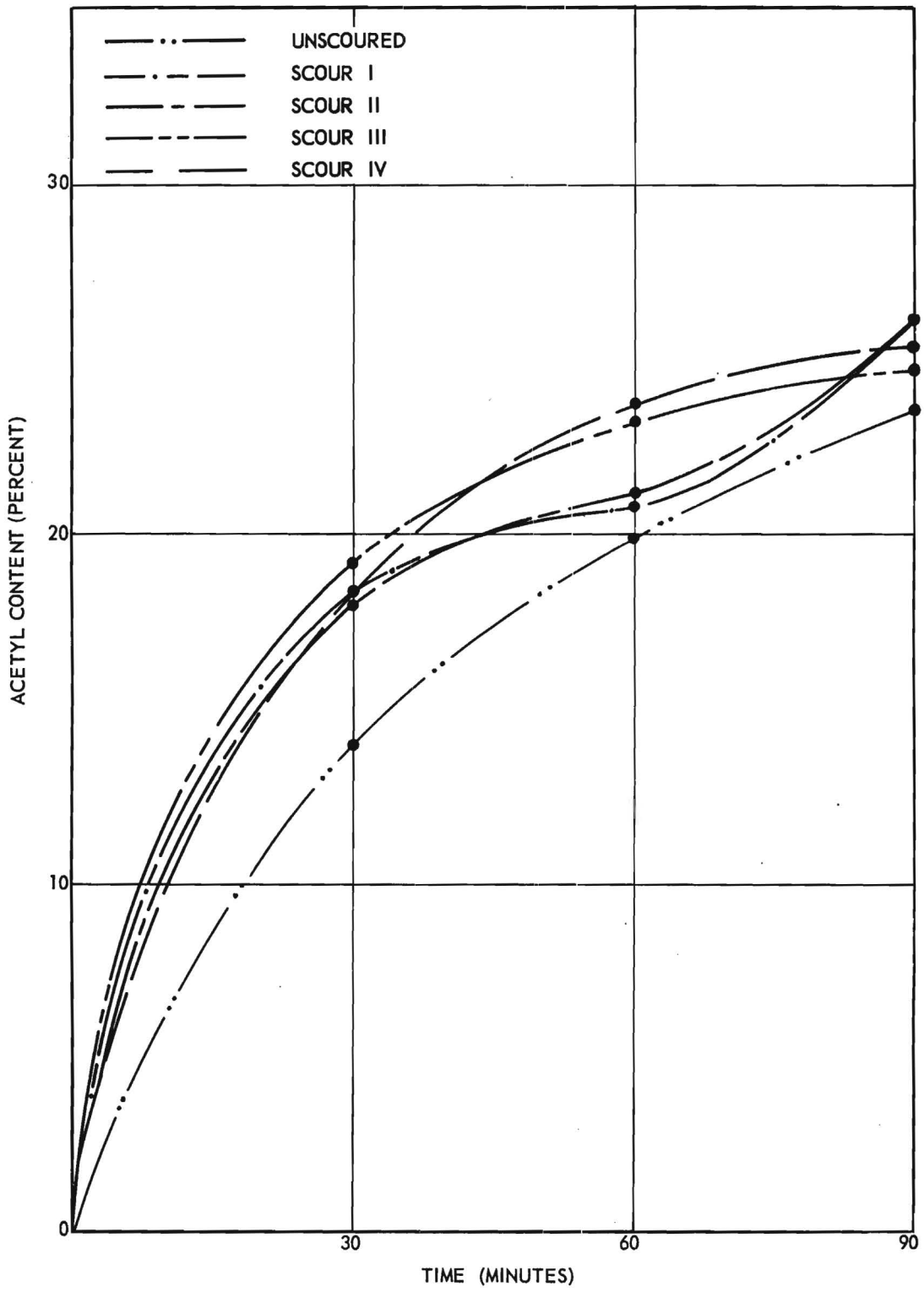

Figure 6. The Change of Acetyl Content With Time of Acetylation for Memphis Cotton Unscoured and Scoured by Four Methods.

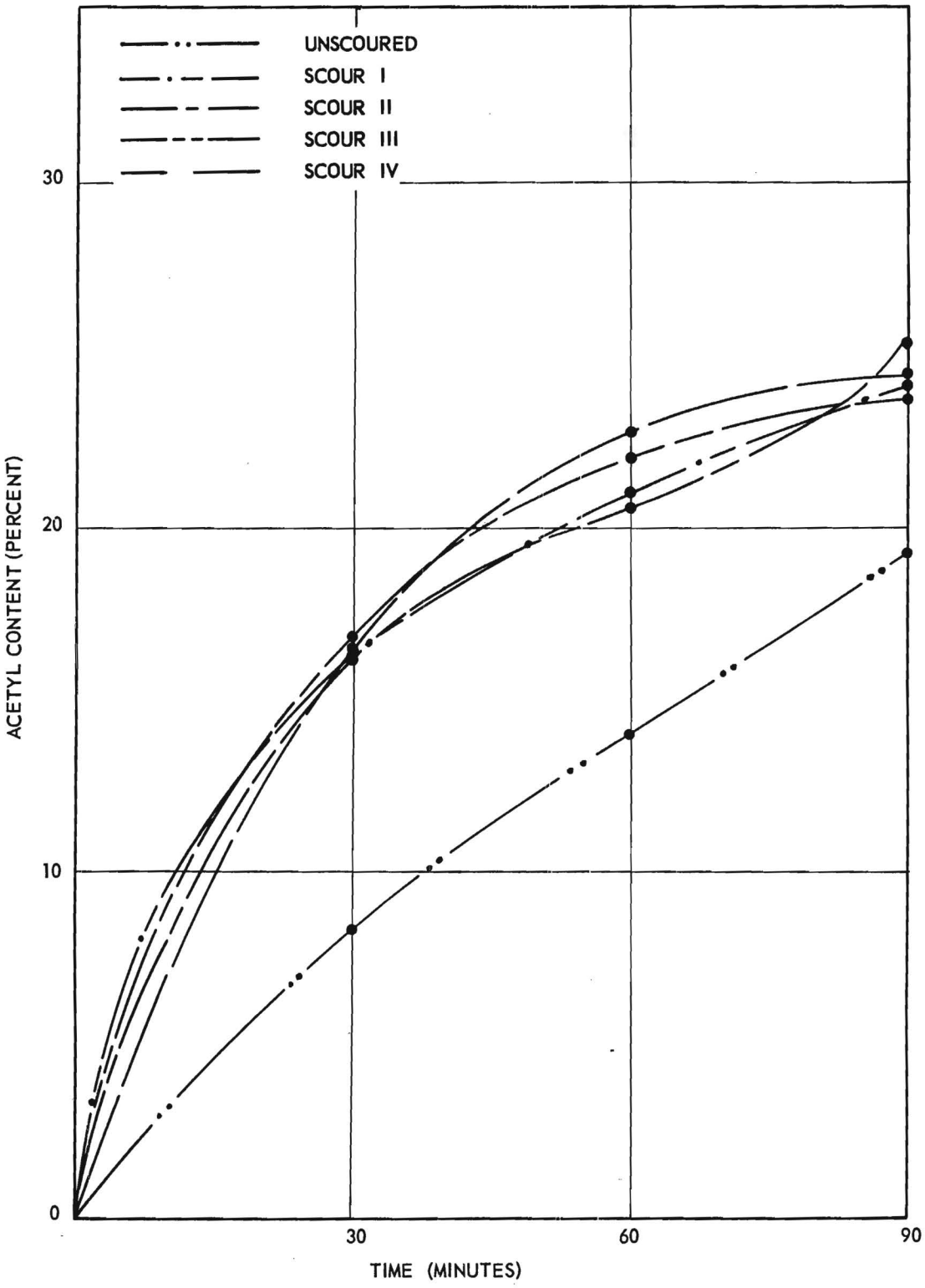

Figure 7. The Change of Acetyl Content With Time of Acetylation for Empire Bale 92 Cotton Unscoured and Scoured by Four Methods.

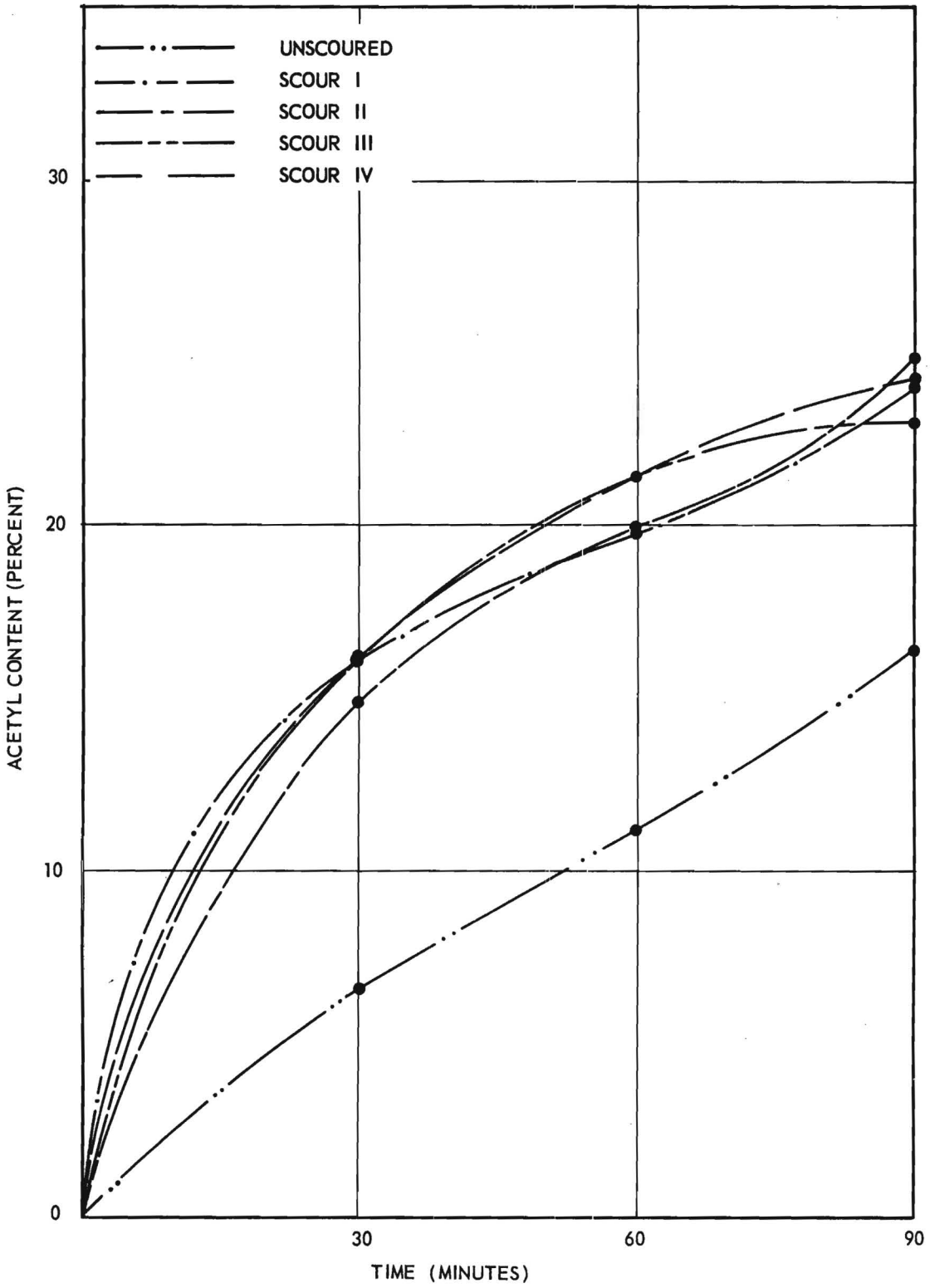

Figure 8. The Change of Acetyl Content With Time of Acetylation for Bob Shaw Cotton Unscoured and Scoured by Four Methods.

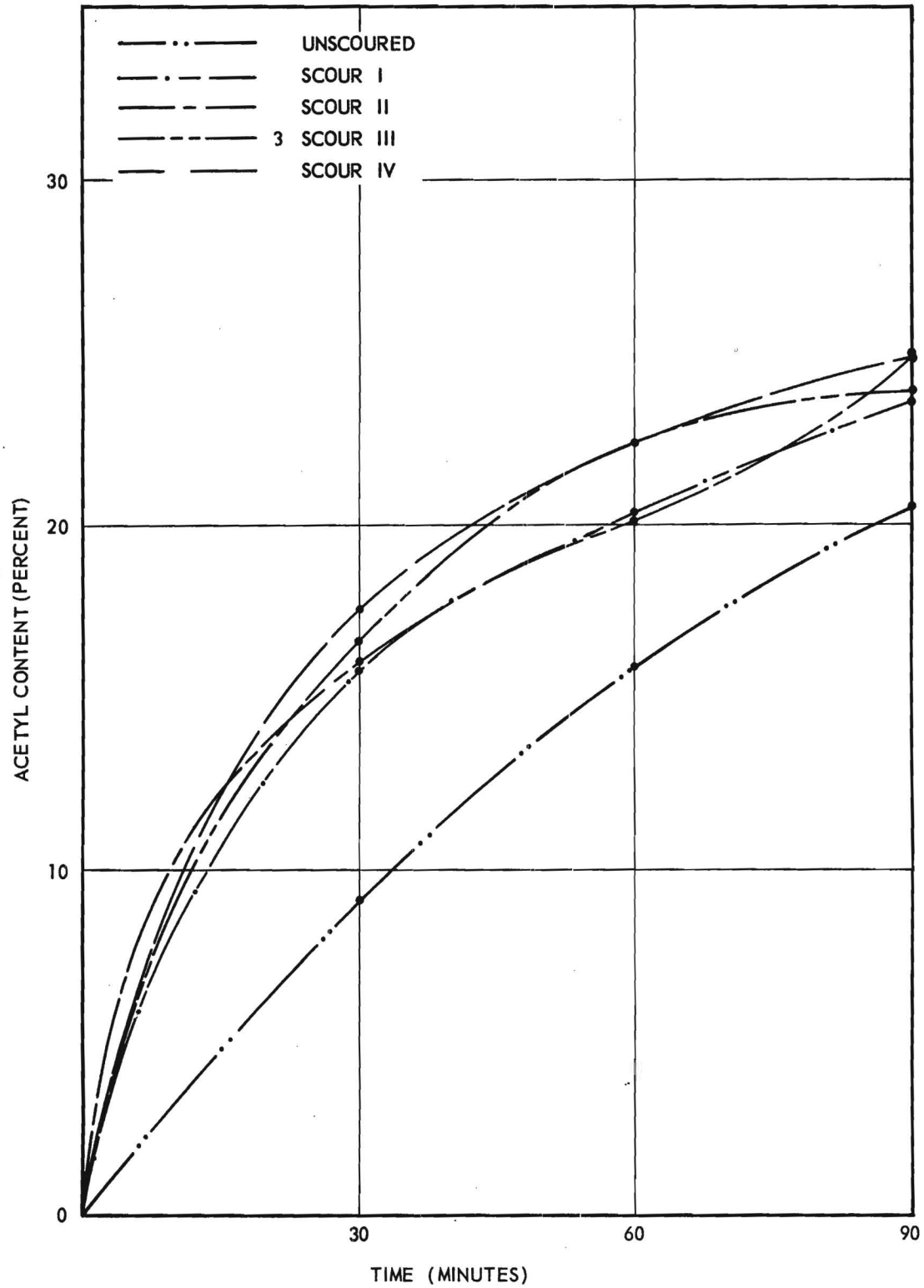

Figure 9. The Change of Acetyl Content With Time of Acetylation for Stoneville 2B Bale 249290 Cotton Unscoured and Scoured by Four Methods.

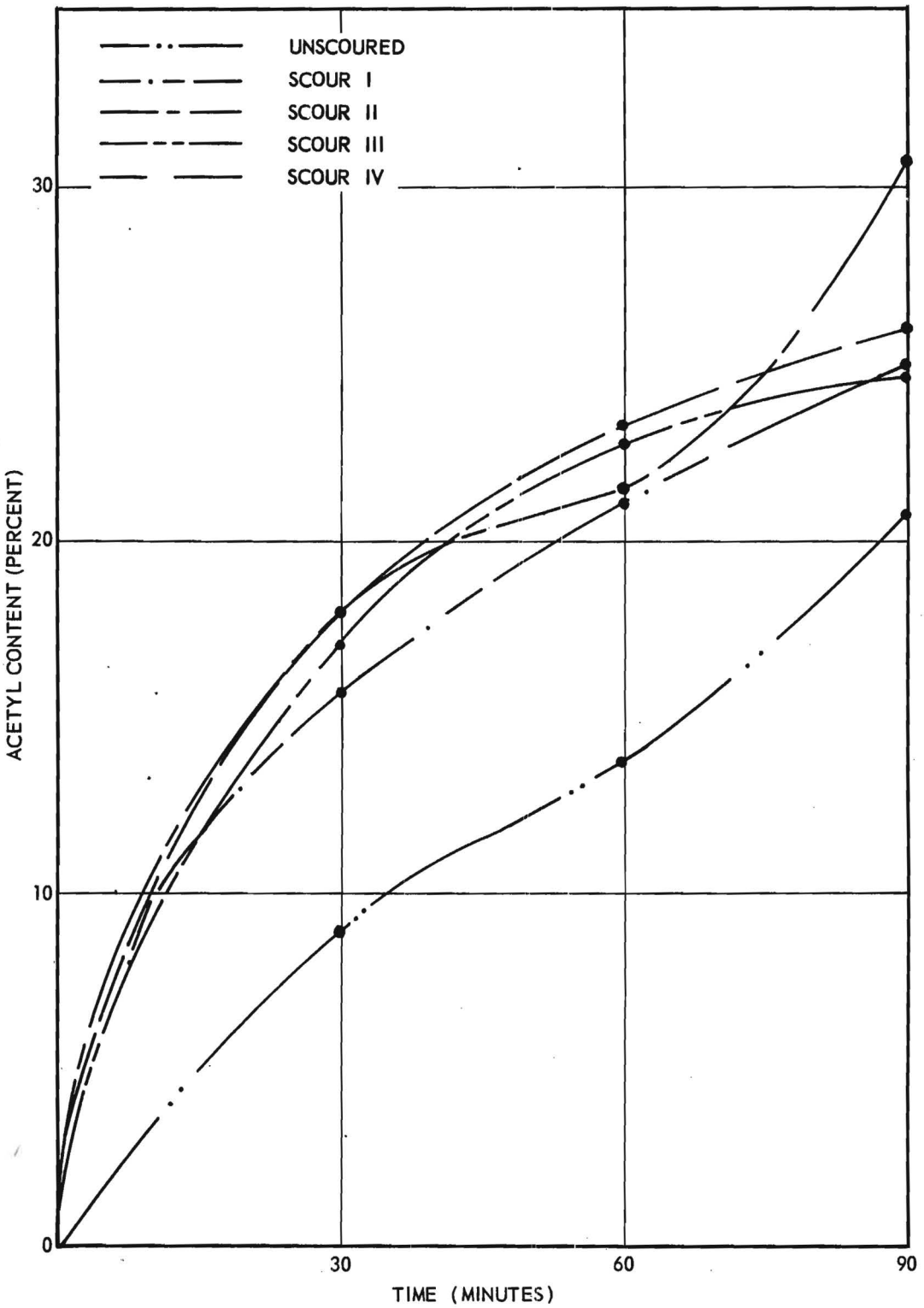

Figure 10. The Change of Acetyl Content With Time of Acetylation for Acala 1517 Cotton Unscoured and Scoured by Four Methods.

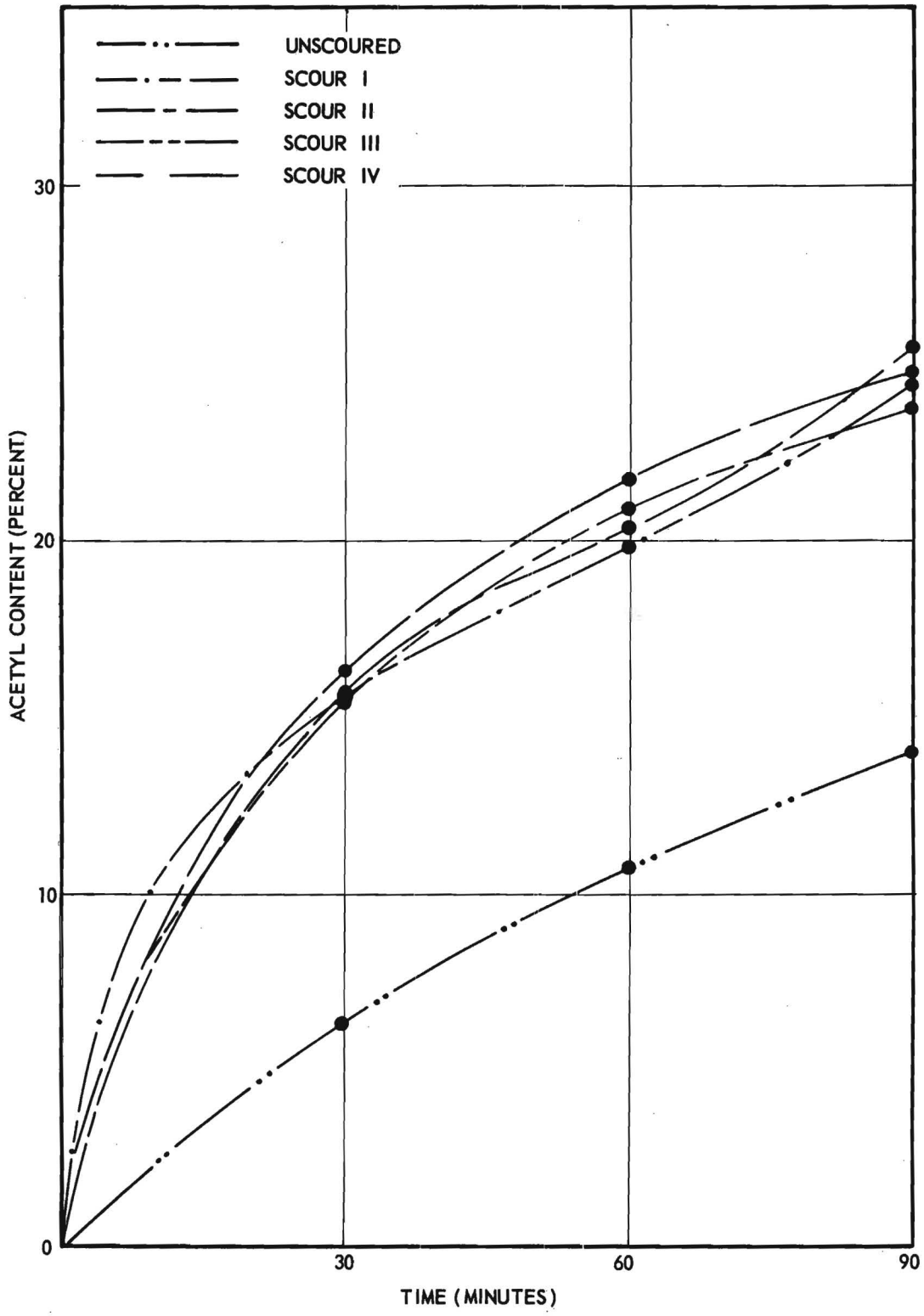

Figure 11. The Change of Acetyl Content With Time of Acetylation for Lockett 140 Cotton Unscoured and Scoured by Four Methods.

samples are presented in Table III. Because of operating errors the data on the 10- and 60-minute runs were discarded and will be repeated and reported later with the remainder of the analyses.

III. DISCUSSION OF RESULTS

The results of the investigations carried out during this period indicate that there is a definite relationship between maturity, fiber fineness and rate of acetylation. The acetyl content of the immature Memphis cotton is consistently higher than any other cottons acetylated in this group. The two most mature cottons, Bob Shaw and Lockett 140, appear to be the least responsive to acetylation treatments. This is particularly true for the unscoured samples. It should be noted that the Micronaire readings of Bob Shaw and Lockett 140 are the highest of this group of cottons.

Although the maturity of the three remaining cottons range from 72 to 86 per cent, very little difference can be observed in their reaction toward acetylation treatments.

Scouring prior to acetylation increases the rate of acetylation for all cottons. This is especially noticeable in the mature cottons. Scouring also tends to minimize the difference in the acetylation of various cottons. An examination of the 60-minute acetylation of unscoured cotton reveals a variation from 10.9 to 20.0 per cent in acetyl content, while for the same period of acetylation in Method IV there is only a small difference in the acetyl content of all cottons; the percentage ranged from 21.5 to 23.7.

TABLE III

ANALYTICAL DATA FOR ACETYLATED COTTONS AT VARIOUS PRESOAKING TIMES AT 100° F

Cottons*	0 Minutes			30 Minutes			120 Minutes			240 Minutes		
	Acetyl (%)	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire	Acetyl (%)	Moisture (%)	Micronaire
Memphis	3.72	5.8	2.60	14.30	2.70	2.70	18.80	2.60	2.75	19.50	4.00	2.75
Empire Bale 92	1.53	5.4	3.95	8.72	3.60	3.78	11.80	3.20	3.87	13.70	4.00	3.92
Bob Shaw	0.87	5.4	5.43	4.70	3.30	5.07	8.45	3.60	5.27	9.20	4.90	5.43
Stoneville 2B Bale 249290	1.37	5.7	3.80	8.05	3.50	3.70	13.80	3.20	3.73	15.30	3.90	3.83
Acala 1517	1.43	5.7	4.27	6.50	4.20	4.17	14.30	3.30	4.23	17.60	3.80	4.35
Lockett 140	1.02	5.8	6.03	4.89	3.60	5.95	9.06	3.50	5.93	11.00	4.40	5.96

*Cottons acetylated at 64° F for 45 minutes.

It should also be noted that as the acetyl content increases the Micronaire readings increase slightly, while the moisture content of the fibers descreases. The moisture content of the scoured acetylated samples is considerably lower than that of unscoured acetylated samples having the same acetyl content.

Partial results of the study of the effect of temperatures at time of presoaking with glacial acetic acid is presented in Table III and Figure 12. Since the analyses of all of the acetylations have not been completed, very little discussion of the results can be made at this time. Complete analyses and discussion will be presented in a later report. Although acetylations have been made on cottons conditioned at 15 per cent R. H. and 65 per cent R. H., analyses of these acetylations have not been completed and will also be presented when completed.

IV. FUTURE PROGRAM

Work will continue on the study of the effect of moisture content on the rate and degree of acetylation of the selected cottons.

The study of the effect of time and temperature of presoaking in glacial acetic acid will also be completed. From these data, an optimum time and temperature of presoaking will be selected for further study of the effects of temperature on the rate and degree of acetylation.

Respectfully submitted:

James L. Taylor
Project Director

Approved:

Herschel H. Cudd, Acting Director
Engineering Experiment Station

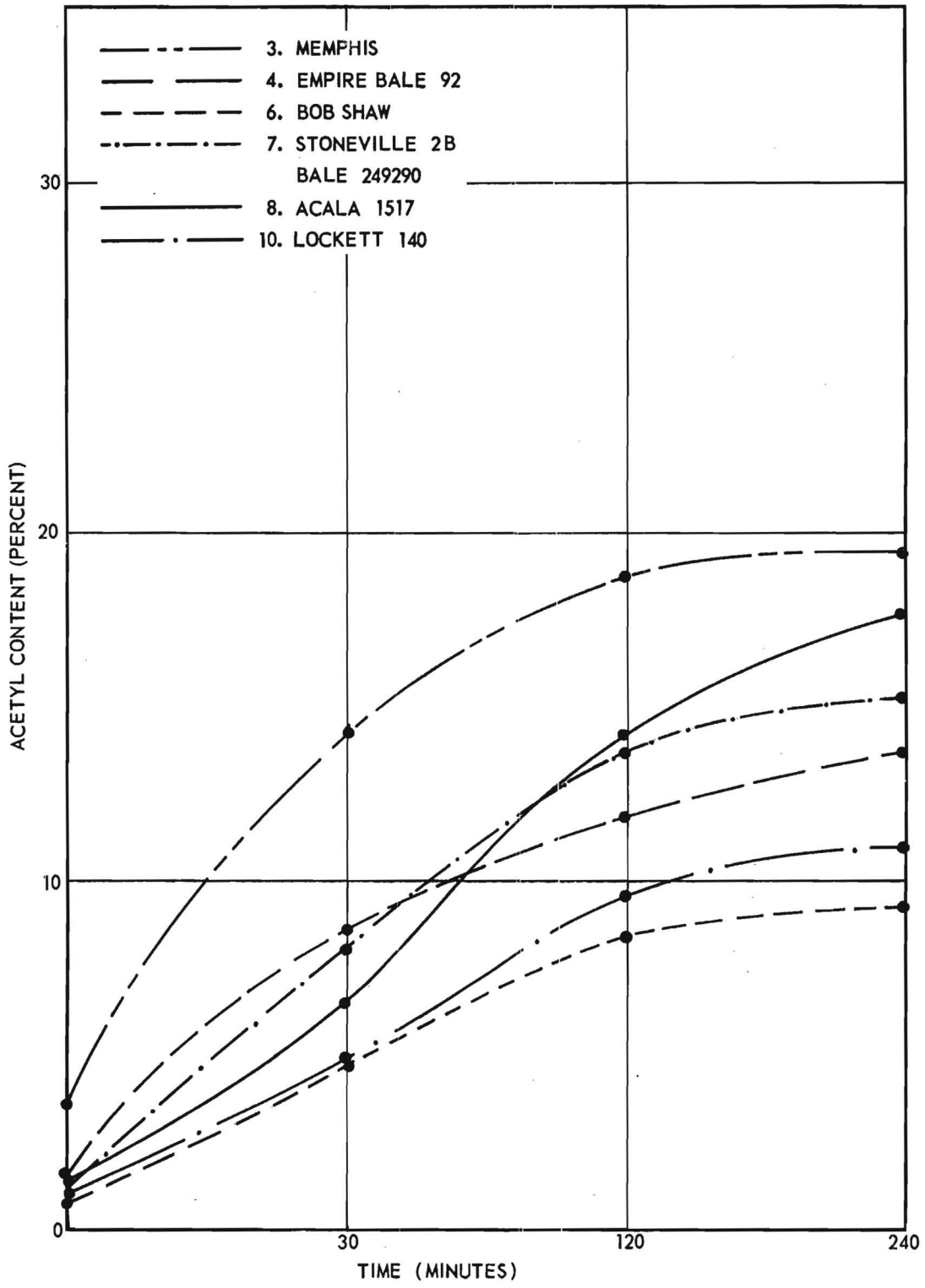

Figure 12. The Change in Acetyl Content With Presoaking Time at 100° F.

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 5

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

JUNE 20, 1953

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 5

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

JUNE 20, 1953

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES

LIST OF FIGURES

This Report Contains 48 Pages

I. WORK PROGRAM

The modified acetylation machine described in Quarterly Report No. 4 (Figures 1 and 2)* was used in the following work program conducted during the past quarter.

A. A study has been made on the effect of moisture conditioning ranging from 15 to 85 per cent relative humidity at 70° F. on the rate and degree of acetylation of six selected varieties of cotton. These cottons are the same as those reported in Progress Report No. 4.

B. In the study of the effect of presoaking on degree of acetylation, the six cottons were presoaked at various times ranging from 10 minutes to 240 minutes, while the temperature of presoaking varied from 70° F. to 170° F.

C. An investigation of the effect of temperature on the degree of acetylation at constant presoak conditions of 10 minutes at 170° F. was completed. The temperature of acetylation varied from 58° to 82° F., while the time for all acetylations was 45 minutes.

D. Moisture content, fiber strength and fiber fineness were determined to show the effect of acetylation on the physical properties of the various cottons.

- - - - -

*Tables and figures are presented in the Appendix.

II. EXPERIMENTAL WORK

A. The Effect of Conditioning at Various Relative Humidities on Acetylation

The moisture conditioning tests which were started in the previous quarter were completed and the results are shown in Tables I through III, and Figures 3 through 14. The samples were conditioned in a desiccator which contained a sulfuric acid solution adjusted to give 15, 35, 50 and 85 per cent relative humidity. The samples were presoaked 18 hours and then acetylated at 64° F. for 30, 60 and 90 minutes using the same procedure described in Progress Report No. 4 (Section B, page 3).

B. The Effect of Presoaking Time and Temperature on the Degree of Acetylation

The six cottons were presoaked in glacial acetic acid for 10, 30, 60, 120 and 240 minutes at 70°, 100°, 130° and 170° F. All samples were acetylated for 45 minutes at 64° F. The results of these experiments are presented in Tables IV through VII and Figures 15 through 29.

C. The Effect of the Temperature of Acetylation on the Degree of Acetylation

Constant presoaking conditions of 170° F. for 10 minutes were selected for this study. The six varieties of cotton were then acetylated for 45 minutes at 58°, 64°, 70°, 76° and 82° F., respectively. These results are presented in Table VIII and Figure 30.

). The Effect of Acetylation on the Physical Properties of Cotton

After acetylation, the cottons were tested for (1) fiber strength using he Pressley Fiber Strenth Tester, (2) fiber fineness by the Sheffield icronaire Machine, and (3) per cent moisture as determined by the Brabender oisture Tester. Tables I through VIII and Figures 31 and 32 show the esults of these tests. All physical tests were carried out at standard aboratory conditions of 70° F. and 65 per cent relative humidity.

III. DISCUSSION OF RESULTS

An examination of the results of moisture conditioning, as shown in Figures 3 through 13, shows that increasing moisture contents in the wide range corresponding to 35 to 85 per cent relative humidity at 70° F. increases the rate and the degree of acetylation for all the cottons under investigation.

The data indicate that the mature cottons are more affected by the moisture conditioning than are the immature varieties. For example, Lockett 40 cotton exhibits a steady, almost linear, rise in degree of acetylation while increasing moisture content at 60 minutes acetylation time. The rise is less regular and not as pronounced for the immature Memphis variety and the Acala 1517 variety, suggesting that immaturity and moisture conditions during growth of the cottons alter the effect of moisture conditioning prior to acetylation.

For all varieties of cotton there is no appreciable difference in rate or degree of acetylation produced by conditioning at 15 per cent and 35 per cent relative humidity. This may be explained by assuming that the degree of swelling produced by conditioning at 35 per cent relative humidity or below is compensated for by the presoak in glacial acetic acid.

The cottons conditioned at a relative humidity greater than 35 per cent exhibit a greater swelling than that produced by glacial acetic acid and therefore exhibit a greater rate and degree of acetylation.

The data presented in Tables IV through VII and Figures 15 through show that increasing the presoaking temperature at constant time sults in a rapid increase in the degree of acetylation until a maximum

acetyl content is reached; then further increases in presoaking temperature produce no higher acetyl contents. At the 100° F. presoaking temperature the maximum per cent acetyl was obtained at a presoak time of 120 minutes; at 130° F. approximately 60 minutes presoak were required for maximum per cent acetyl; and at 170° F. only 10 minutes presoak was needed to reach maximum acetyl. This statement holds for all the cottons except for the very immature Memphis variety, which reached its maximum acetyl content with a presoak time of 10 minutes at 100° F., 130° F. and 170° F.

The maximum acetyl content was never reached for the 70° F. presoak within the time range established for these presoaking experiments.

The results of the studies to show the effect of acetylation temperature on the degree of acetylation are shown in Table VIII and Figure 30. From these data it is apparent than an increase of 24° F. (58° to 82° F.) in the acetylation temperature approximately doubled the acetyl content for the various cottons. For example, the immature Memphis cotton increased in acetyl content from 12.77 to 23.7 per cent. The mature Pockett 140 cotton increased from 7.96 to 19.9 per cent and the intermediately mature cotton, Stoneville 2B, increased from 10.7 to 21.5 per cent. Over the entire range of acetylation temperatures, 58° F., 64° F., 70° F., 76° F. and 82° F., the increase in acetyl content for all the cottons tested was relatively constant, as shown by the straight lines in Figure 30.

In general, the determination of the effect of acetylation on the physical properties of the cottons showed, with increasing acetyl contents, a general decrease in tensile strength, a marked decrease in moisture

content at standard conditions, and a slight swelling or increase in fiber diameter. As illustrated in Figures 31 and 32, the notable exception to the general statement is the marked decrease in fiber strength with increase in acetylation temperature. This degradation was so great at 82° F. acetylation temperature that reliable tensile strengths could not be determined on any variety of cotton.

IV. FUTURE PROGRAM

Work on determination of the rate and degree of acetylation of the original twelve varieties of cotton, along with some others received recently from Southern Regional Research Laboratory, will be concluded during the next quarter. Efforts will also be directed toward determination of the physical and chemical properties of these cottons and relating these results to the rate and degree of acetylation. This should complete Part I and Part II of the subject contract.

Respectfully submitted:

James L. Taylor
Project Director

Approved:

Herschel H. Cudd, Acting Director
Engineering Experiment Station

V. APPENDIX

TABLE I

ANALYTICAL DATA FOR COTTONS ACETYLATED FOR 30 MINUTES AT 64° F.
AFTER CONDITIONING AT VARIOUS RELATIVE HUMIDITIES

Cottons	15 % Relative Humidity				35 % Relative Humidity				50 % Relative Humidity			
	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index
Memphis	12.30	5.4	2.67	6.56	12.20	5.2	2.67	6.53	12.6	5.6	2.60	7.02
Empire Bale 92	5.70	6.2	4.02	7.14	5.39	5.6	3.93	7.44	6.31	6.3	3.93	7.06
Bob Shaw	3.87	6.5	5.38	8.07	3.57	5.4	5.33	8.47	4.47	6.4	5.47	8.49
Stoneville 2B Bale 249290	6.59	5.9	3.92	8.19	6.54	5.7	3.80	8.25	7.64	6.1	3.75	8.21
Acala 1517	6.51	6.1	4.30	8.68	6.90	5.9	4.25	8.34	8.25	6.2	4.25	8.20
Lockett 140	3.40	6.4	6.00	7.40	3.51	5.8	6.05	6.84	3.90	6.3	6.00	7.84

Cottons	65 % Relative Humidity				85 % Relative Humidity			
	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index
Memphis	14.0	4.6	2.67	6.59	14.3	4.7	2.55	6.18
Empire Bale 92	8.00	5.1	3.90	6.83	8.26	5.6	3.90	9.08
Bob Shaw	5.95	5.3	5.38	8.22	5.76	5.6	5.27	8.37
Stoneville 2B Bale 249290	9.37	5.0	3.80	7.80	9.81	5.4	3.68	8.14
Acala 1517	7.95	5.2	4.20	8.70	11.4	5.7	4.20	8.48
Lockett 140	6.32	5.4	5.95	7.05	6.84	5.9	5.93	7.06

TABLE II

ANALYTICAL DATA FOR COTTONS ACETYLATED FOR 60 MINUTES AT 64° F.
AFTER CONDITIONING AT VARIOUS RELATIVE HUMIDITIES

Cottons	15 % Relative Humidity				35 % Relative Humidity				50 % Relative Humidity			
	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index
Memphis	18.4	4.6	2.75	6.13	17.6	4.5	2.68	6.14	19.4	4.4	2.73	6.13
Empire Bale 92	10.6	5.6	3.93	6.92	9.67	5.7	3.97	6.87	11.7	5.0	4.02	6.73
Bob Shaw	7.59	5.2	5.33	8.07	6.89	5.1	5.33	8.28	8.82	4.8	5.38	8.17
Stoneville 2B Bale 249290	11.70	5.2	3.80	7.51	10.7	5.3	3.93	8.01	13.0	5.2	3.82	7.91
Acala 1517	11.30	5.4	4.33	8.21	11.8	5.3	4.27	8.36	14.0	5.4	4.52	8.02
Lockett 140	6.84	5.8	6.00	7.12	6.87	6.0	5.98	7.18	8.51	5.5	5.95	7.16

Cottons	65 % Relative Humidity				85 % Relative Humidity			
	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index
Memphis	19.5	4.1	2.80	6.14	21.2	4.2	2.73	6.11
Empire Bale 92	13.5	4.4	4.02	6.47	14.7	4.3	4.00	6.79
Bob Shaw	10.6	5.0	5.43	7.80	11.3	4.8	5.50	8.19
Stoneville 2B Bale 249290	15.1	4.5	3.85	7.17	16.3	4.6	3.80	7.37
Acala 1517	14.0	4.9	4.33	7.76	19.1	4.4	4.33	7.36
Lockett 140	10.8	4.9	5.95	6.65	12.2	4.9	6.07	6.86

Progress Report No. 5, Project No. 208-156

TABLE III

ANALYTICAL DATA FOR COTTONS ACETYLATED FOR 90 MINUTES AT 64° F.
AFTER CONDITIONING AT VARIOUS RELATIVE HUMIDITIES

Cottons	15 % Relative Humidity				35 % Relative Humidity				50 % Relative Humidity			
	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index
Memphis	20.7	3.9	3.35	5.45	20.9	3.9	3.22	5.81	24.3	4.1	3.23	5.73
Empire Bale 92	15.9	4.2	4.43	5.98	15.3	4.4	4.25	6.63	17.5	4.6	4.43	6.11
Bob Shaw	12.2	4.1	5.70	7.35	12.6	4.6	5.53	7.86	14.6	4.8	5.78	7.34
Stoneville 2B Bale 249290	16.8	3.8	4.28	6.91	17.1	4.2	4.10	7.35	19.2	4.3	4.22	6.90
Acala 1517	17.0	4.2	4.95	7.08	17.3	4.4	4.67	7.11	19.7	4.7	4.90	7.01
Lockett 140	11.7	4.6	6.38	6.79	11.4	5.3	6.30	7.14	13.0	5.2	6.17	6.58

Cottons	65 % Relative Humidity				85 % Relative Humidity			
	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (µg/in.)	Pressley Index
Memphis	23.5	4.7	3.02	5.93	24.8	4.1	3.08	5.66
Empire Bale 92	18.8	4.7	4.30	5.99	18.6	4.4	4.20	6.56
Bob Shaw	15.6	4.9	5.52	7.26	15.6	4.1	5.55	7.49
Stoneville 2B Bale 249290	19.2	4.4	4.10	6.34	20.4	4.1	4.08	6.77
Acala 1517	20.3	4.8	4.55	6.74	22.9	4.0	4.88	6.94
Lockett 140	13.5	5.2	6.22	6.51	16.2	4.5	6.13	6.55

TABLE IV

ANALYTICAL DATA FOR ACETYLATED COTTONS AT VARIOUS PRESOAKING TIMES AT 70° F.

Cottons	0 Minutes				10 Minutes				30 Minutes			
	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index
Memphis	3.72	5.8	2.60	6.24	8.07	5.8	2.62	6.87	10.5	5.6	2.63	6.68
Empire Bale 92	1.53	5.4	3.95	6.99	2.75	5.7	3.87	7.16	3.89	5.7	4.00	6.92
Bob Shaw	.87	5.4	5.43	8.11	1.46	5.5	5.30	8.20	2.36	5.5	5.37	8.13
Stoneville 2B Bale 249290	1.37	5.7	3.80	7.71	3.37	5.8	3.66	8.16	3.97	5.7	3.83	7.87
Acala 1517	1.43	5.7	4.27	8.63	2.17	5.8	4.20	8.32	2.78	5.8	4.30	8.11
Lockett 140	1.02	5.8	6.03	7.05	1.37	5.9	5.92	7.32	1.84	5.9	6.05	7.20

Cottons	60 Minutes				120 Minutes				240 Minutes			
	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index
Memphis	13.1	5.4	2.68	6.44	14.1	4.8	2.68	6.20	14.3	4.4	2.70	6.97
Empire Bale 92	5.07	5.6	3.90	6.69	6.26	5.4	3.90	6.64	7.28	5.1	3.97	6.51
Bob Shaw	3.07	5.4	5.32	7.93	4.38	5.3	5.35	8.07	4.42	4.9	5.40	8.02
Stoneville 2B Bale 249290	4.87	5.6	3.75	7.96	6.80	5.5	3.73	7.73	8.03	5.0	3.83	7.54
Acala 1517	4.20	5.9	4.22	8.11	5.62	5.3	4.25	8.04	6.79	5.3	4.28	8.01
Lockett 140	2.59	5.9	5.95	7.21	2.62	5.7	6.05	6.98	4.04	5.5	6.03	6.84

TABLE V

ANALYTICAL DATA FOR ACETYLATED COTTONS AT VARIOUS PRESOAKING TIMES AT 100° F.

Cottons	0 Minutes				10 Minutes				30 Minutes			
	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index
Memphis	3.72	5.8	2.60	6.24	16.9	4.9	2.80	6.84	15.5	4.7	2.70	6.37
Empire Bale 92	1.53	5.4	3.95	6.99	7.83	5.3	3.90	6.55	8.91	4.7	3.78	6.80
Bob Shaw	.87	5.4	5.43	8.11	4.62	5.0	5.35	8.33	4.59	5.3	5.07	8.42
Stoneville 2B Bale 249290	1.37	5.7	3.80	7.71	7.90	5.4	3.80	7.13	8.23	5.1	3.70	7.09
Acala 1517	1.43	5.7	4.27	8.63	6.61	5.8	4.30	8.27	6.44	5.6	4.17	8.50
Lockett 140	1.02	5.8	6.03	7.05	4.33	5.2	5.90	7.43	4.74	5.3	5.95	6.96

Cottons	60 Minutes				120 Minutes				240 Minutes			
	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index
Memphis	17.2	4.4	2.82	6.10	18.1	4.0	2.75	5.91	18.5	4.0	2.75	5.70
Empire Bale 92	9.86	5.1	4.10	6.52	11.8	4.6	3.87	6.13	13.0	4.8	3.92	5.98
Bob Shaw	6.44	4.9	5.35	8.07	8.45	5.2	5.27	7.68	9.26	4.9	5.43	7.18
Stoneville 2B Bale 249290	11.5	5.0	3.88	6.93	13.8	4.2	3.73	6.68	14.8	3.9	3.83	5.99
Acala 1517	9.75	5.3	4.30	7.84	14.5	4.4	4.23	7.32	17.6	3.8	4.35	7.28
Lockett 140	5.45	5.5	5.95	6.99	9.06	5.1	5.93	6.85	10.6	4.4	5.96	6.48

TABLE VI

ANALYTICAL DATA FOR ACETYLATED COTTONS AT VARIOUS PRESOAKING TIMES AT 130° F.

Cottons	0 Minutes				10 Minutes				30 Minutes			
	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index
Memphis	3.72	5.8	2.60	6.24	17.3	4.6	2.85	6.86	17.1	4.1	2.85	6.78
Empire Bale 92	1.53	5.4	3.95	6.99	8.43	4.8	4.02	6.90	10.3	4.7	4.00	6.38
Bob Shaw	.87	5.4	5.43	8.11	6.08	5.2	5.47	8.08	8.30	4.9	5.40	7.82
Stoneville 2B Bale 249290	1.37	5.7	3.80	7.71	11.6	4.8	3.90	7.33	12.6	4.5	3.91	7.00
Acala 1517	1.43	5.7	4.27	8.63	9.81	4.3	4.35	8.19	12.4	4.5	4.33	7.64
Lockett 140	1.02	5.8	6.03	7.05	6.21	5.2	5.97	7.50	7.42	5.0	6.00	6.97

Cottons	60 Minutes				120 Minutes				240 Minutes			
	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index
Memphis	17.5	3.7	2.87	6.47	19.2	3.8	2.80	6.33	18.6	3.9	2.87	6.05
Empire Bale 92	12.3	4.4	4.13	6.38	13.4	4.0	3.95	6.27	13.6	4.2	4.01	5.91
Bob Shaw	9.87	4.5	5.50	7.74	12.1	4.4	5.22	7.01	12.3	4.3	5.42	6.87
Stoneville 2B Bale 249290	13.9	4.2	3.93	6.95	14.6	4.2	3.80	6.91	14.7	4.1	3.93	6.71
Acala 1517	15.6	4.3	4.45	7.58	16.2	3.9	4.25	7.14	16.4	4.2	4.30	7.04
Lockett 140	9.91	4.5	6.08	6.69	11.1	4.5	6.02	6.70	10.8	4.4	6.18	6.76

TABLE VII

ANALYTICAL DATA FOR ACETYLATED COTTONS AT VARIOUS PRESOAKING TIMES AT 170° F.

Cottons	0 Minutes				10 Minutes				30 Minutes			
	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index	Acetyl (%)	Moisture (%)	Micronaire (μg/in.)	Pressley Index
Memphis	3.72	5.8	2.60	6.24	16.5	4.7	2.63	6.89	17.0	4.7	2.73	6.38
Empire Bale 92	1.53	5.4	3.95	6.99	11.4	5.2	3.93	6.91	12.0	5.1	4.08	6.53
Bob Shaw	.87	5.4	5.43	8.11	8.90	5.4	5.38	7.68	9.53	5.0	5.40	6.95
Stoneville 2B Bale 249290	1.37	5.7	3.80	7.71	12.8	4.8	3.80	6.64	13.0	5.3	4.02	6.21
Acala 1517	1.43	5.7	4.27	8.63	14.0	5.0	4.22	8.29	14.5	5.3	4.42	7.64
Lockett 140	1.02	5.8	6.03	7.05	8.86	5.2	5.90	7.25	9.27	5.3	6.02	6.56

Cottons	60 Minutes				120 Minutes				240 Minutes			
	Acetyl (%)	ture (%)	naire (μg/in.)	Index	Acetyl (%)	ture (%)	naire (μg/in.)	Index	Acetyl (%)	ture (%)	naire (μg/in.)	Index
Memphis	16.6	4.3	2.78	6.16	16.8	4.2	2.73	5.97	16.6	4.3	2.80	5.49
Empire Bale 92	12.4	4.6	4.00	6.12	11.6	4.9	4.10	6.05	12.5	4.5	4.17	5.76
Bob Shaw	9.98	4.9	5.43	6.54	9.95	4.2	5.45	6.13	10.8	4.4	5.40	5.61
Stoneville 2B Bale 249290	13.3	5.2	3.85	5.72	12.5	4.5	4.00	5.98	13.9	4.3	4.10	5.88
Acala 1517	14.4	5.0	4.35	6.35	15.0	4.6	4.35	6.35	15.4	4.3	4.52	6.14
Lockett 140	9.81	5.3	5.97	5.97	9.75	4.8	6.07	5.83	11.4	4.6	6.10	5.40

TABLE VIII

ANALYTICAL DATA FOR ACETYLATED COTTONS AT CONSTANT PRESOAKING CONDITIONS*
AND AT VARIOUS ACETYLATION TEMPERATURES

Cottons	58° F. Acetyl (%)	58° F. Moisture (%)	58° F. Micronaire (µg/in.)	58° F. Pressley Index	64° F. Acetyl (%)	64° F. Moisture (%)	64° F. Micronaire (µg/in.)	64° F. Pressley Index	70° F. Acetyl (%)	70° F. Moisture (%)	70° F. Micronaire (µg/in.)	70° F. Pressley Index
Memphis	12.7	4.9	2.68	6.02	16.5	4.7	2.63	6.89	19.2	4.0	3.00	6.09
Empire Bale 92	9.13	4.9	4.02	6.62	11.4	5.2	3.93	6.91	13.5	4.2	4.33	6.49
Bob Shaw	7.48	5.0	5.37	7.70	8.90	5.4	5.38	6.68	10.7	4.6	5.73	6.84
Stoneville 2B Bale 249290	10.7	5.3	3.65	7.16	12.8	4.8	3.80	6.64	15.4	4.3	4.08	6.80
Acala 1517	10.8	5.3	4.32	8.01	14.0	5.0	4.22	8.29	16.8	4.3	4.58	6.93
Lockett 140	7.96	5.4	6.00	6.53	8.86	5.2	5.90	7.29	11.00	4.3	6.27	6.27

Cottons	76° F. Acetyl (%)	76° F. Moisture (%)	76° F. Micronaire (µg/in.)	76° F. Pressley Index	82° F. Acetyl (%)	82° F. Moisture (%)	82° F. Micronaire (µg/in.)	82° F. Pressley Index
Memphis	20.7	4.2	3.08	5.52	23.7	3.9	3.18	—**
Empire Bale 92	17.1	4.4	4.28	6.16	20.4	4.0	4.50	—**
Bob Shaw	15.3	3.9	5.57	6.33	19.3	3.5	5.87	—**
Stoneville 2B Bale 249290	18.3	4.2	4.10	6.17	21.5	3.7	4.23	—**
Acala 1517	20.0	4.0	4.60	6.83	23.1	3.4	4.78	—**
Lockett 140	14.7	4.4	6.10	6.01	19.9	3.7	6.18	—**

*Cottons presoaked 10 minutes at 170° F.
**Fibers too weak to give reliable breaks.

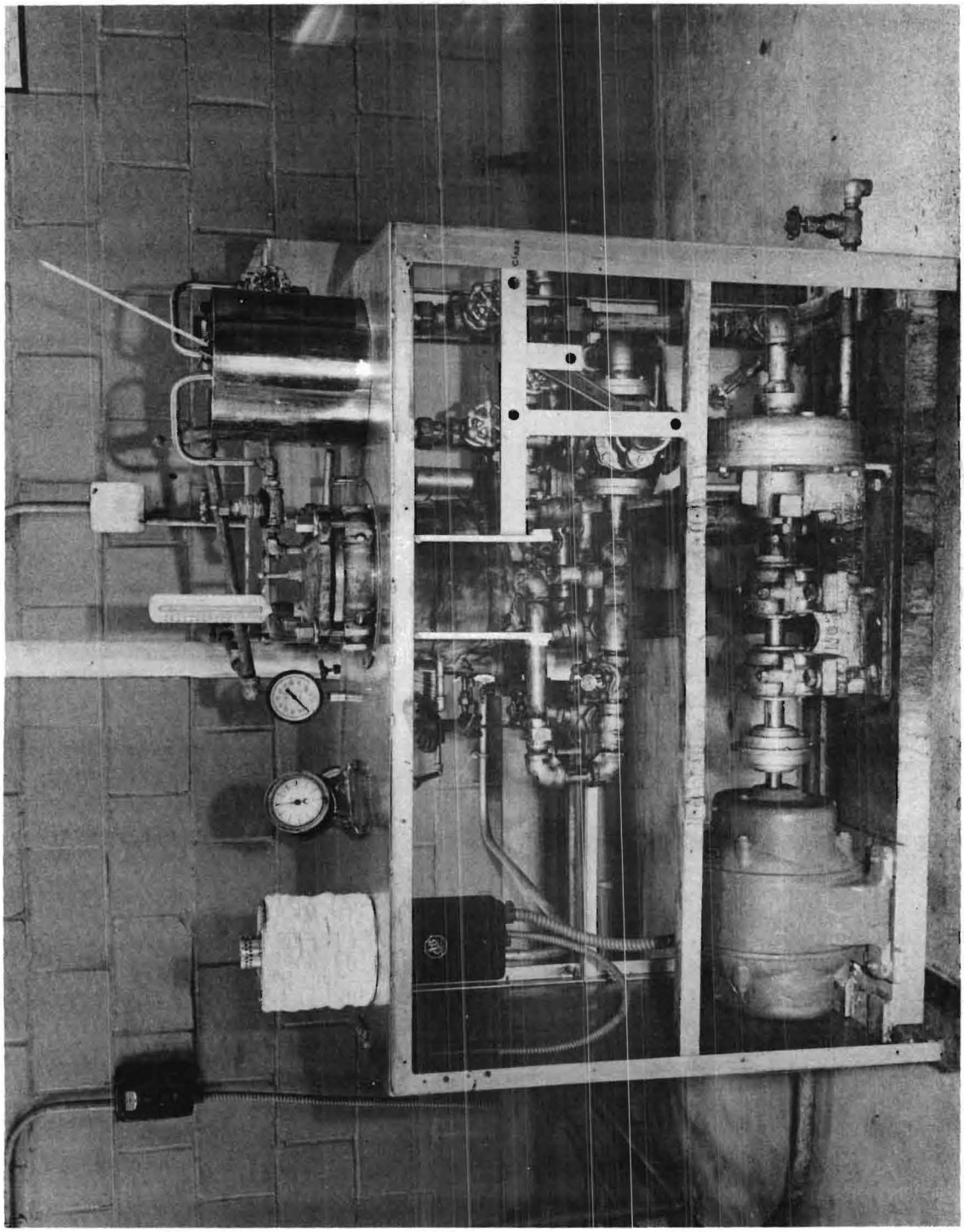

Figure 1. Front View of the Acetylation Machine.

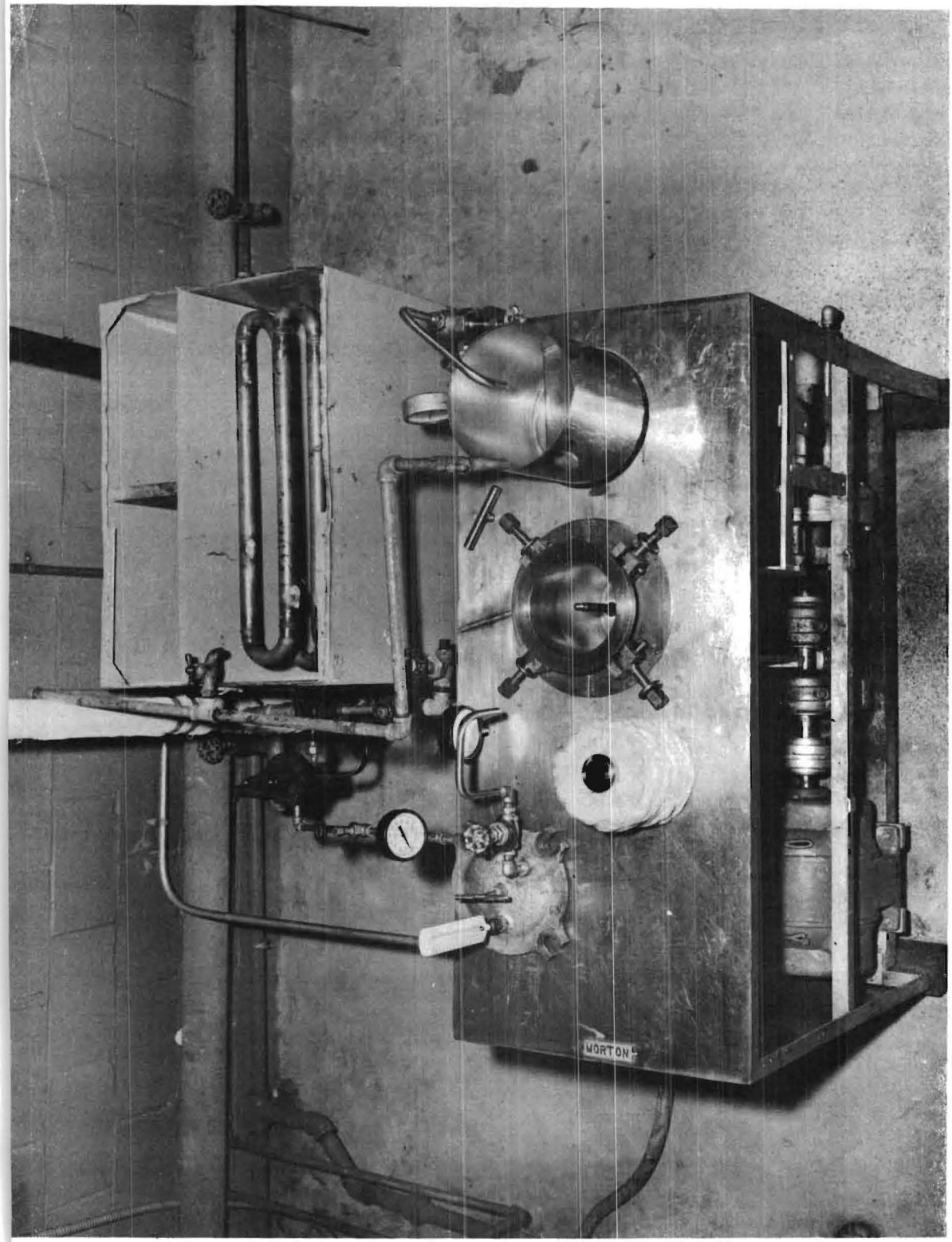

Figure 2. Top View of the Acetylation Machine.

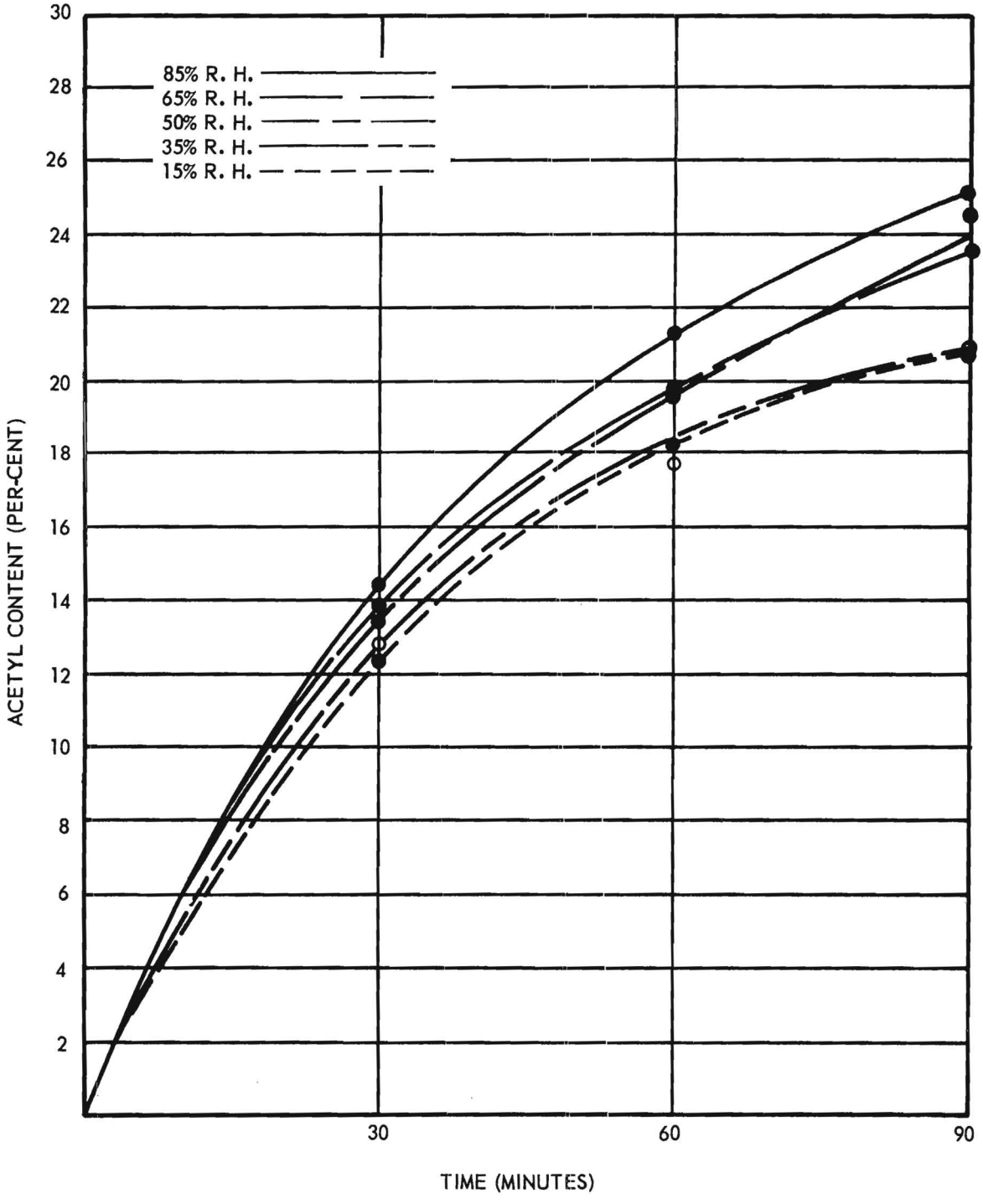

Figure 3. The Change in Acetyl Content with Time of Acetylation for Memphis Cotton Moisture Preconditioned at Five Different Relative Humidities.

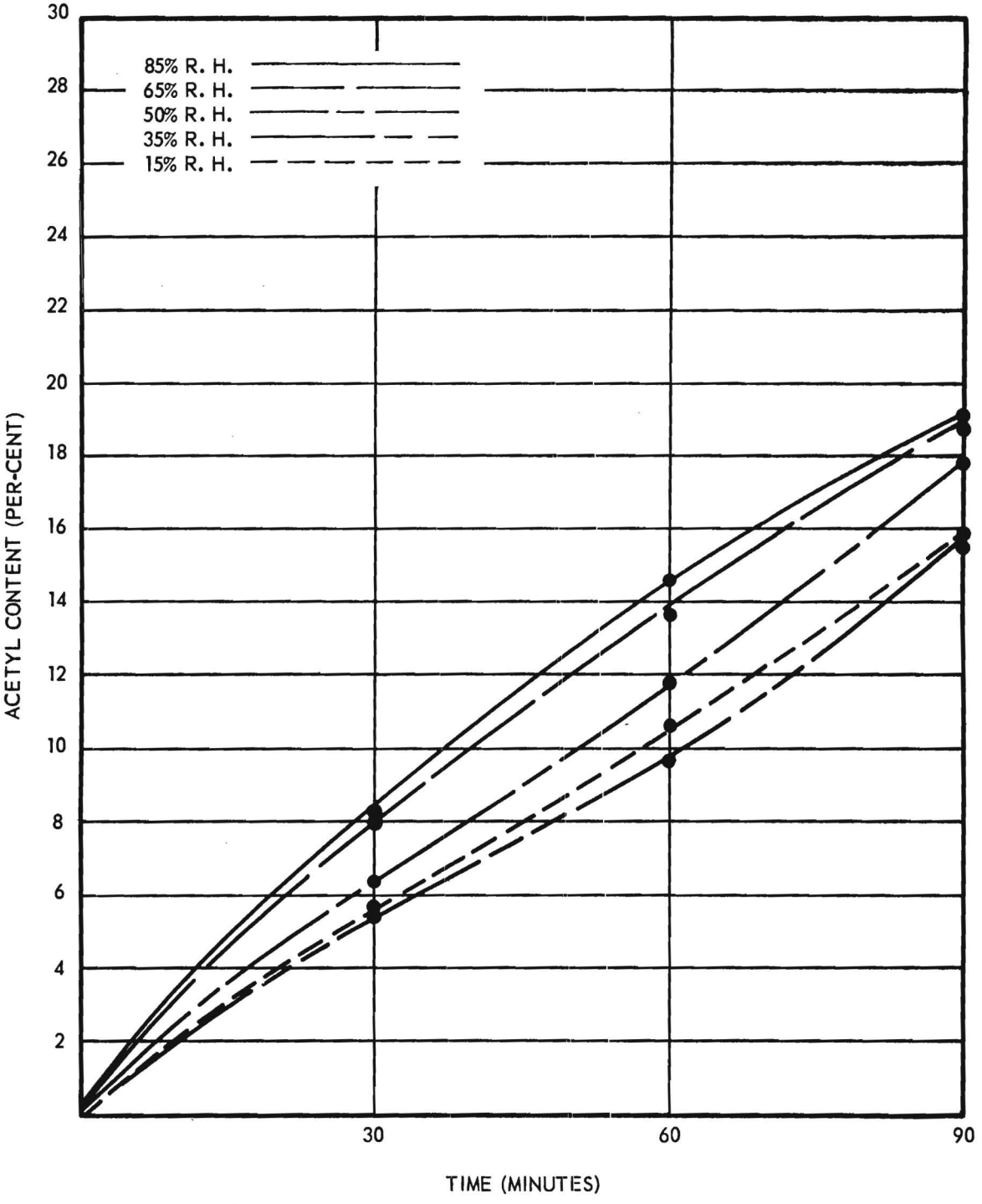

Figure 4. The Change in Acetyl Content with Time of Acetylation for Empire Bale 92 Cotton Moisture Preconditioned at Five Different Relative Humidities.

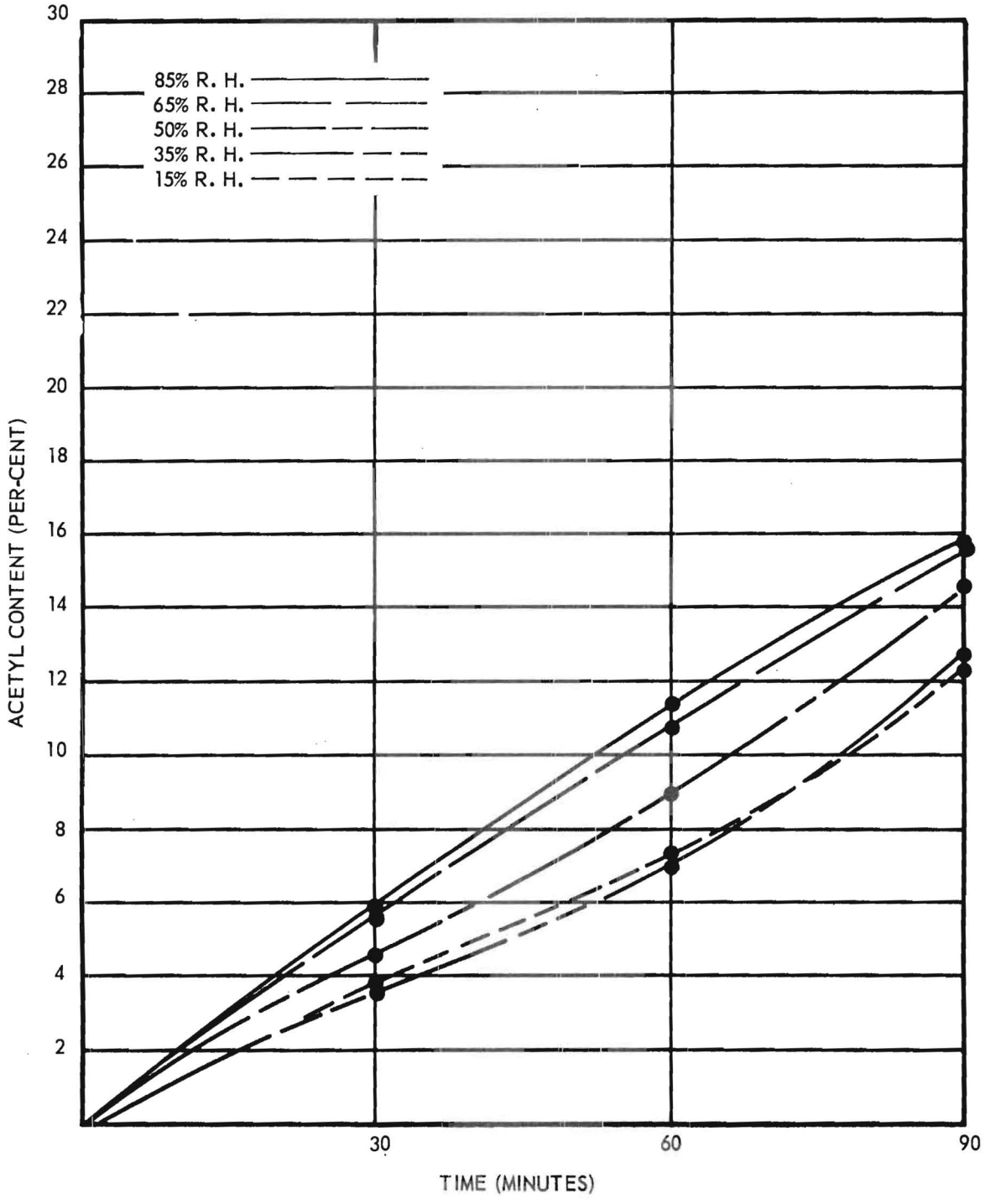

Figure 5. The Change in Acetyl Content with Time of Acetylation for Bob Shaw Cotton Moisture Preconditioned at Five Different Relative Humidities.

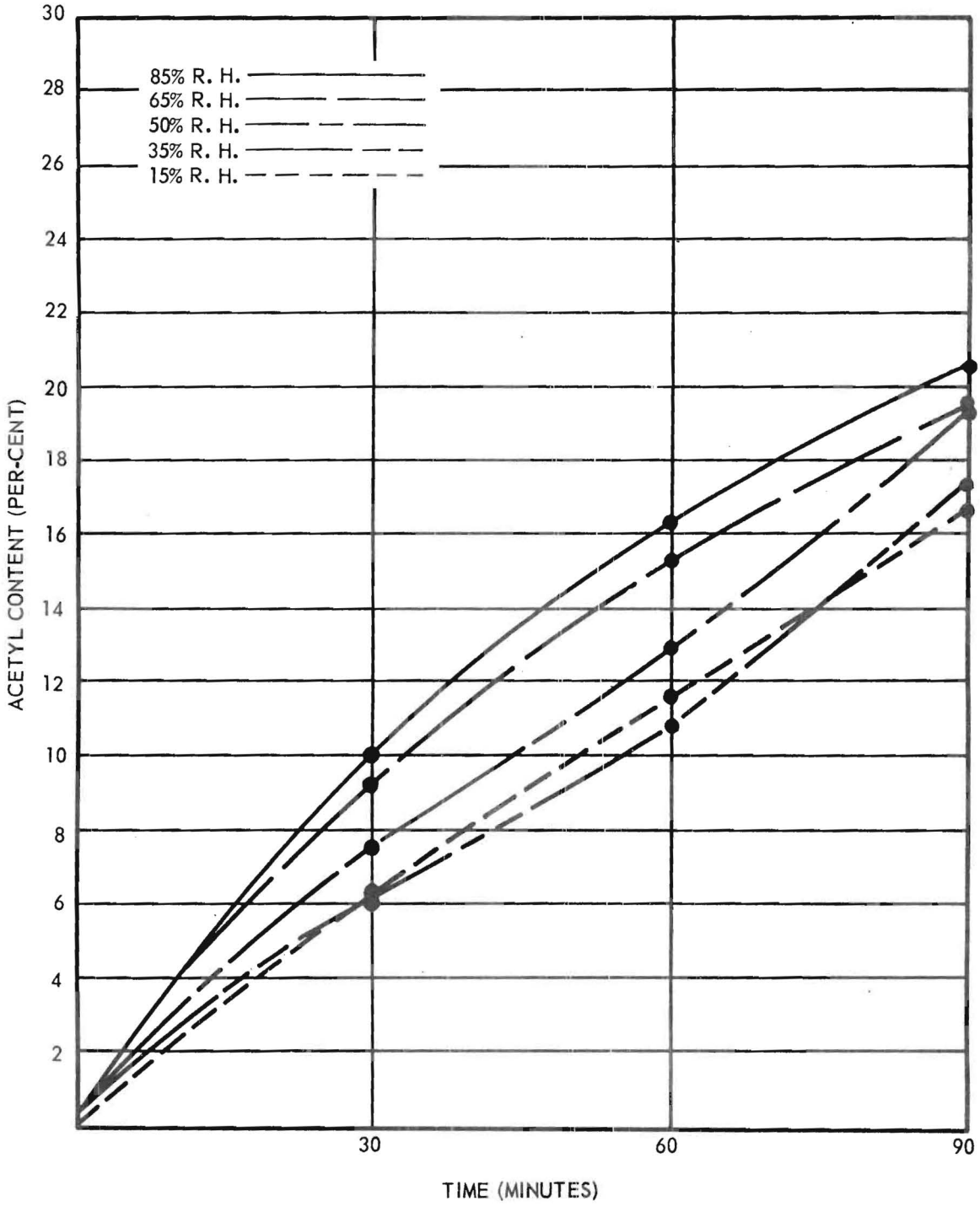

Figure 6. The Change in Acetyl Content with Time of Acetylation for Stoneville 2B Cotton Moisture Preconditioned at Five Different Relative Humidities.

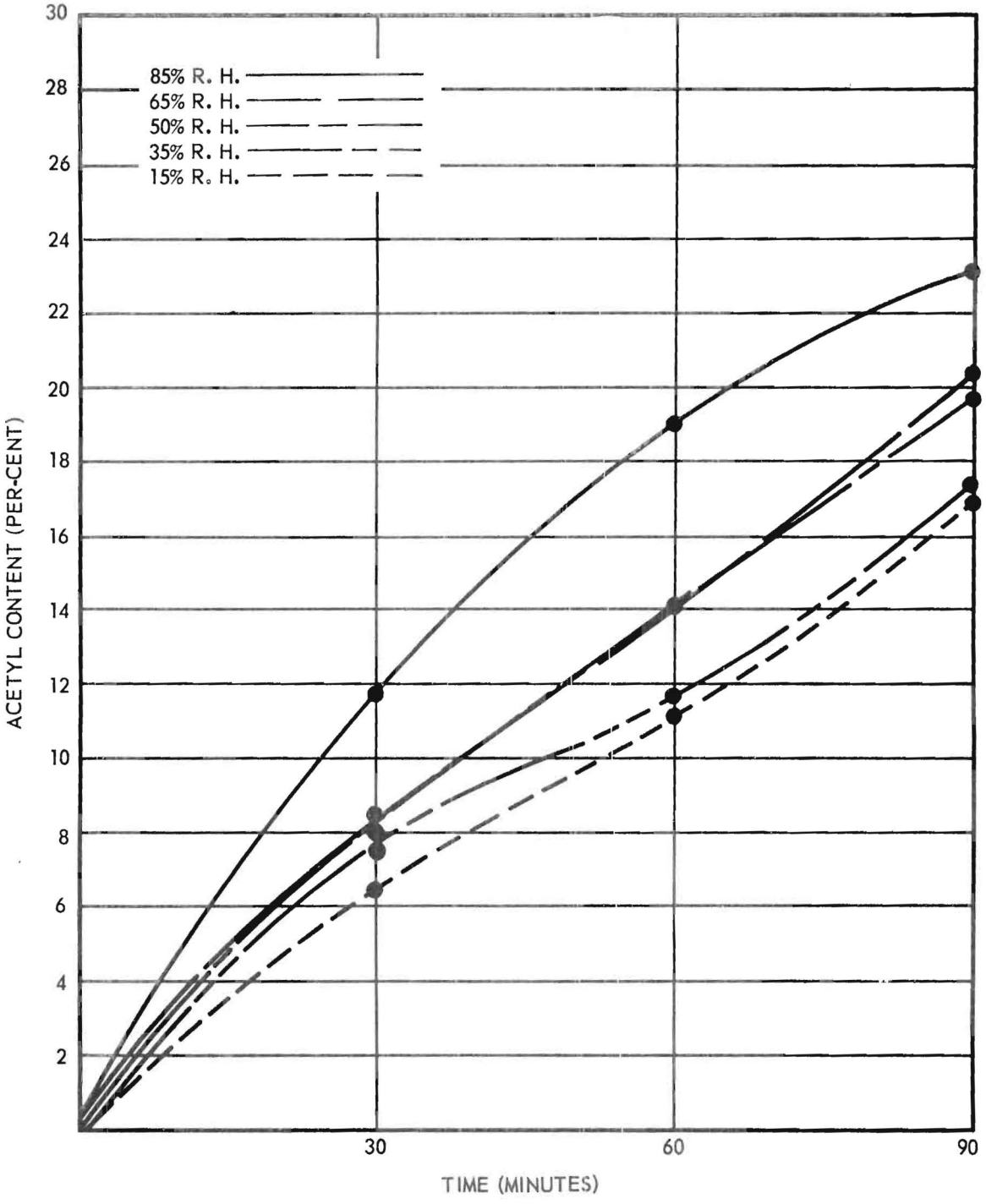

Figure 7. The Change in Acetyl Content with Time of Acetylation for Acala 1517 Cotton Moisture Preconditioned at Five Different Relative Humidities.

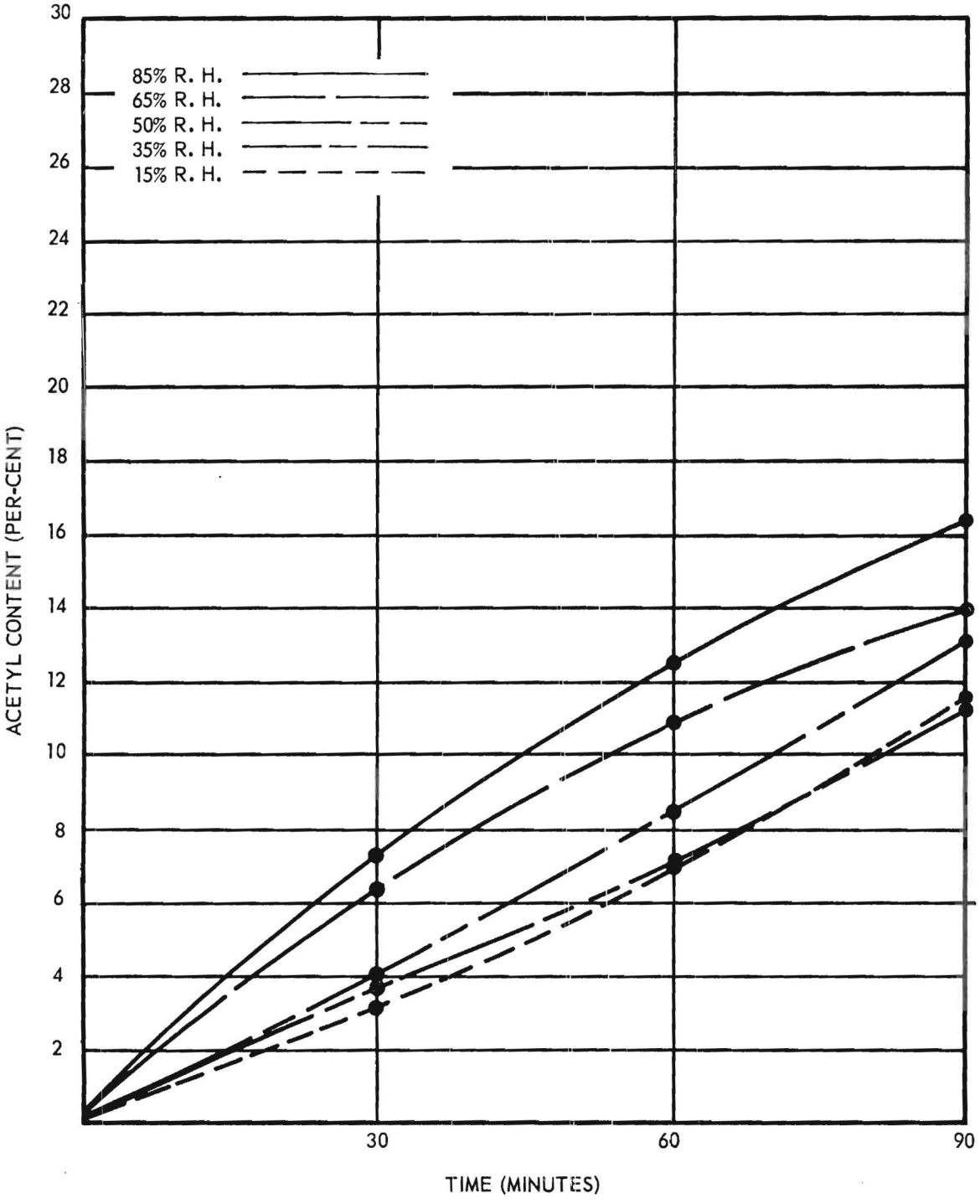

Figure 8. The Change in Acetyl Content with Time of Acetylation for Lockett 140 Cotton Moisture Preconditioned at Five Different Relative Humidities.

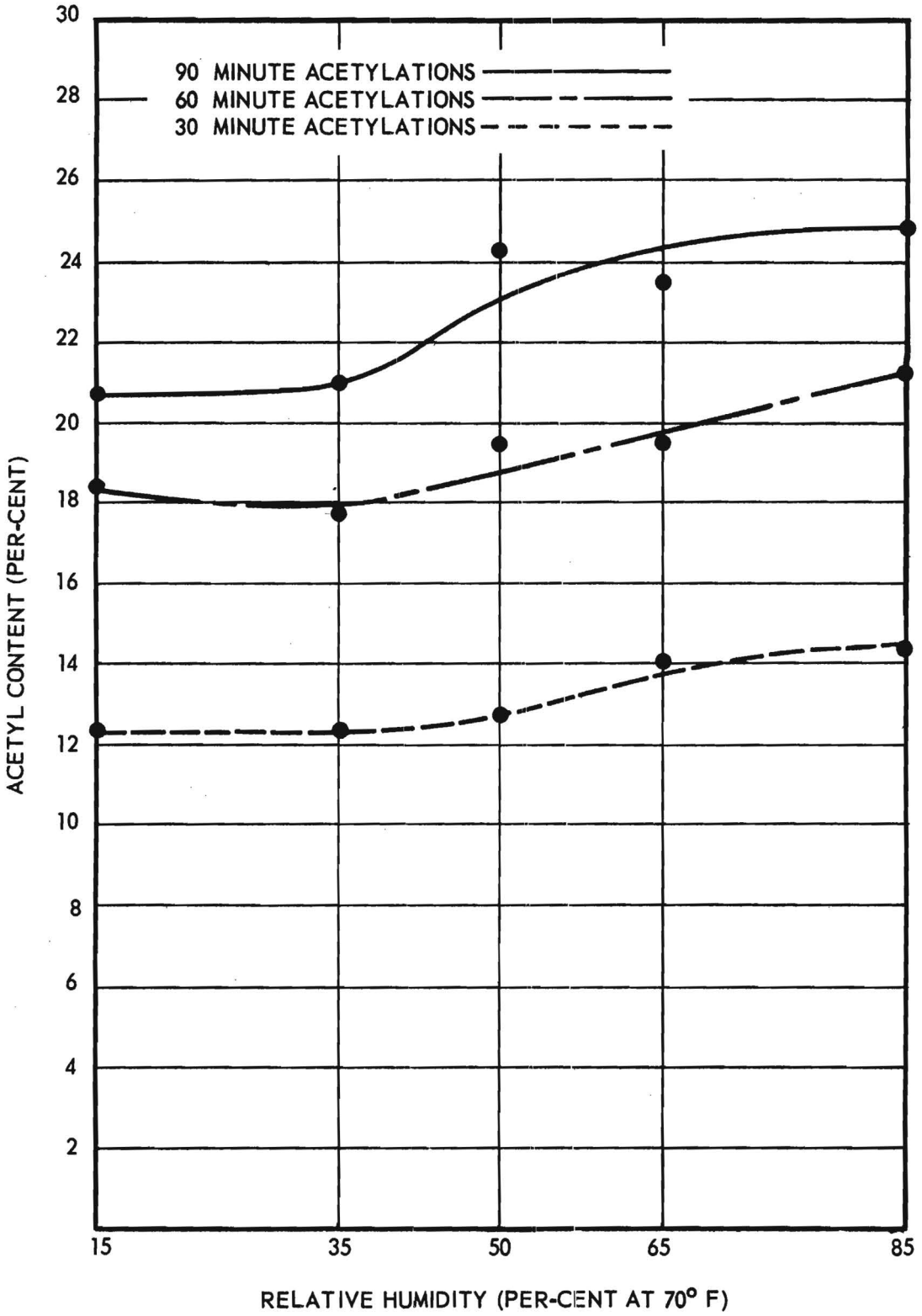

Figure 9. The Change in Acetyl Content with Relative Humidity of Preconditioning for Memphis Cotton at Three Times of Acetylation.

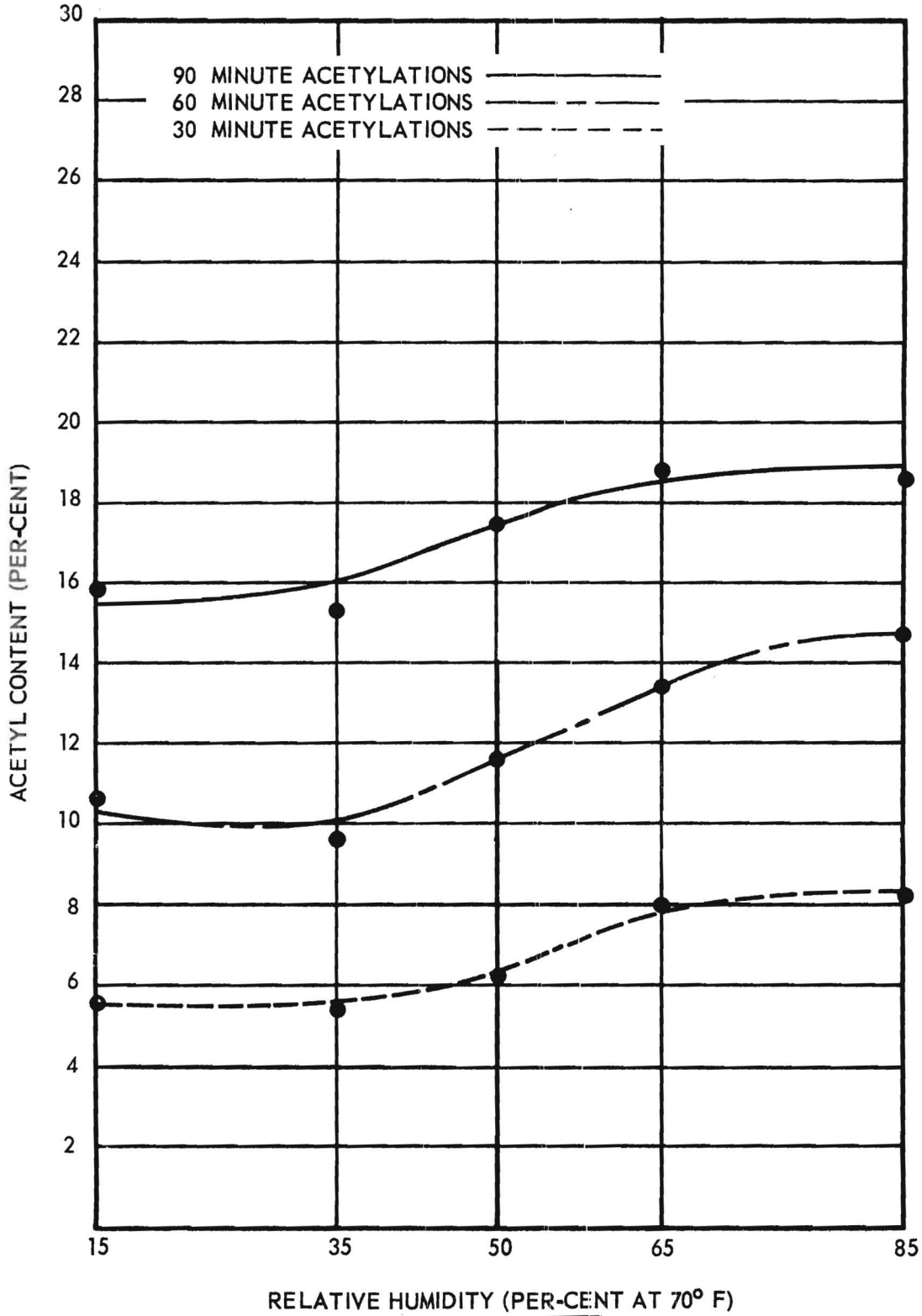

Figure 10. The Change in Acetyl Content with Relative Humidity of Preconditioning for Empire Bale 92 Cotton at Three Times of Acetylation.

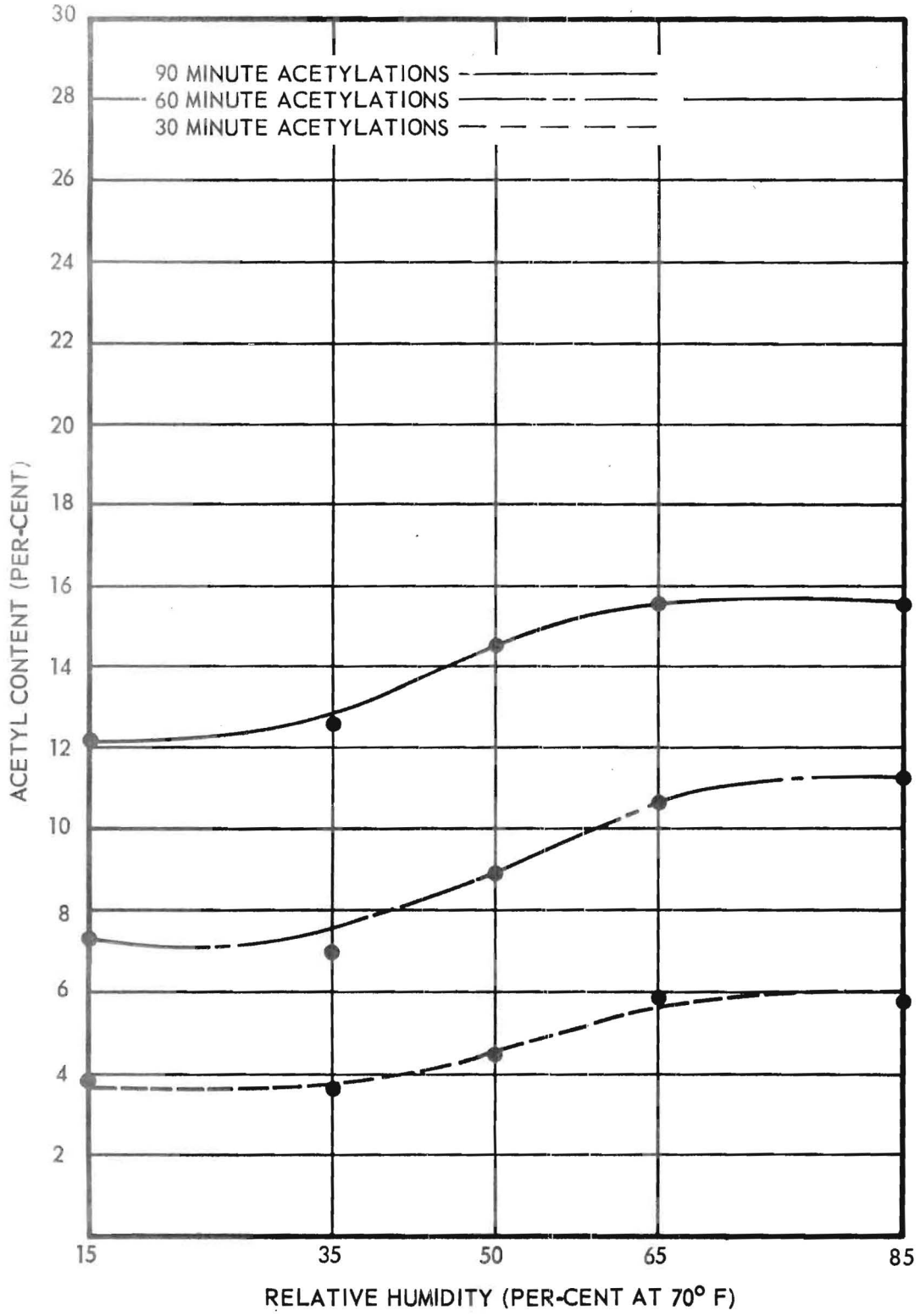

Figure 11. The Change in Acetyl Content with Relative Humidity of Preconditioning for Bob Shaw Cotton at Three Times of Acetylation.

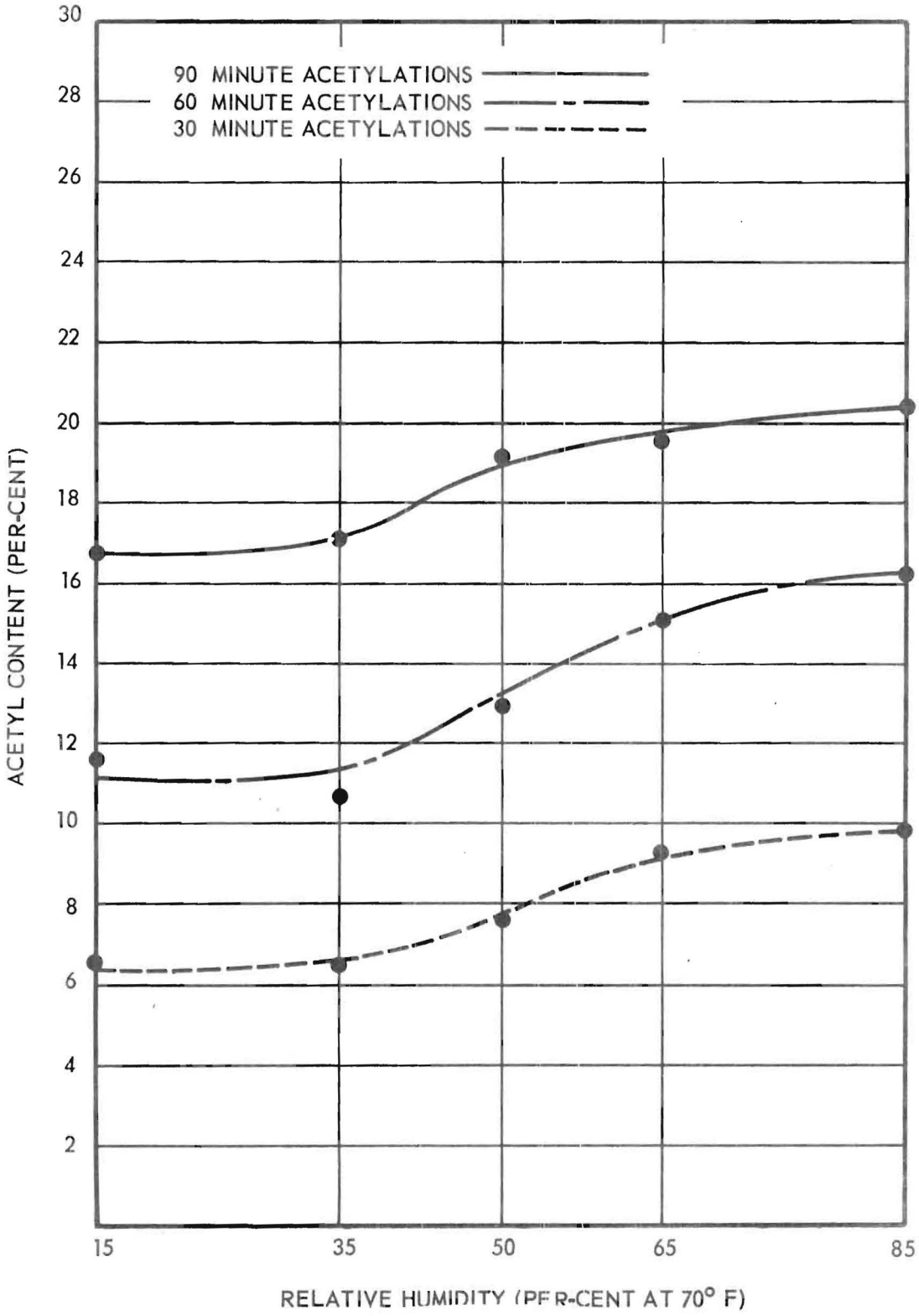

Figure 12. The Change in Acetyl Content with Relative Humidity of Preconditioning for Stoneville 2B Cotton at Three Times of Acetylation.

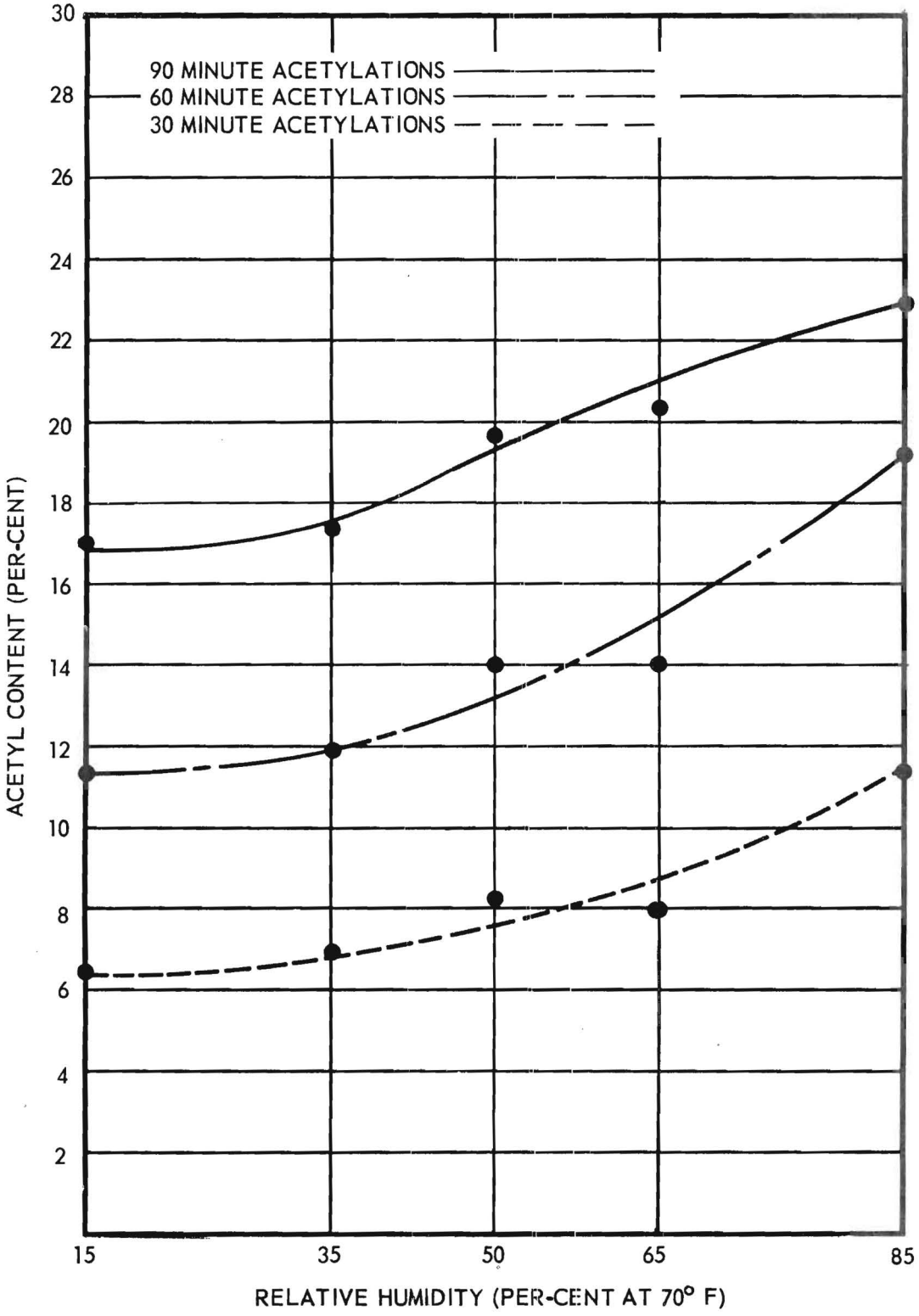

Figure 13. The Change in Acetyl Content with Relative Humidity of Preconditioning for Acala 1517 Cotton at Three Times of Acetylation.

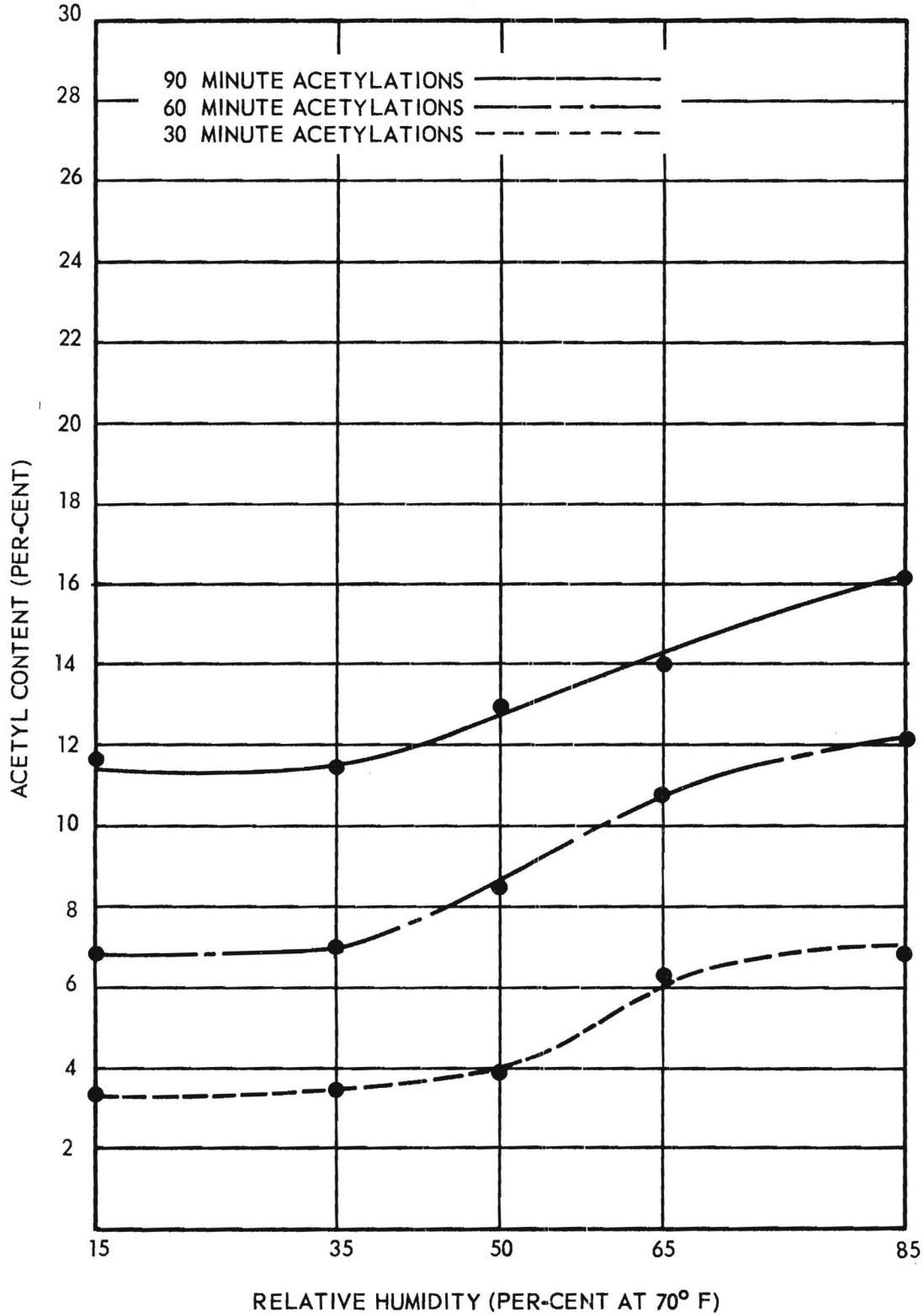

Figure 14. The Change in Acetyl Content with Relative Humidity of Preconditioning for Lockett 140 Cotton at Three Times of Acetylation.

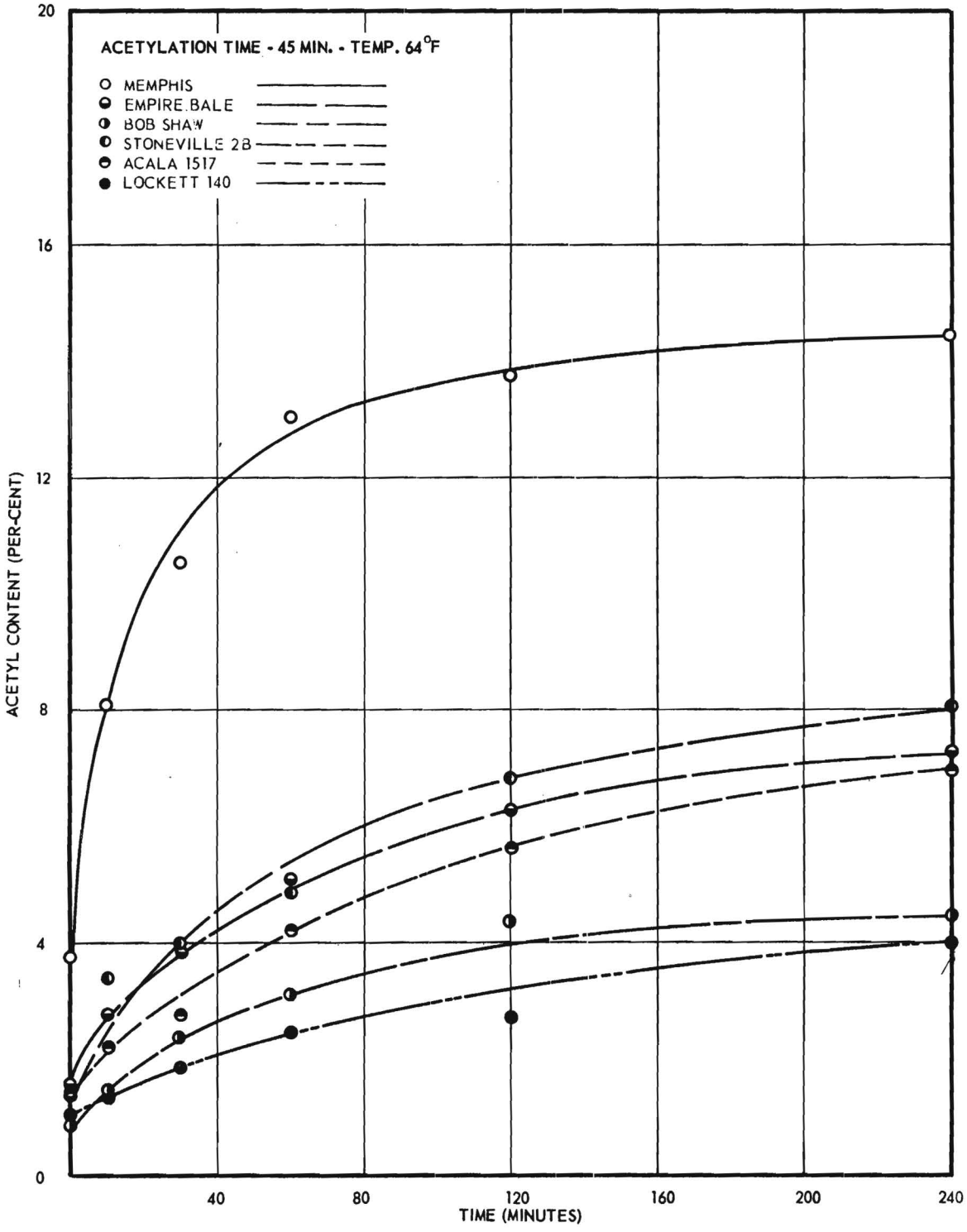

Figure 15. The Change in Acetyl Content with Presoaking Time at 70°F.

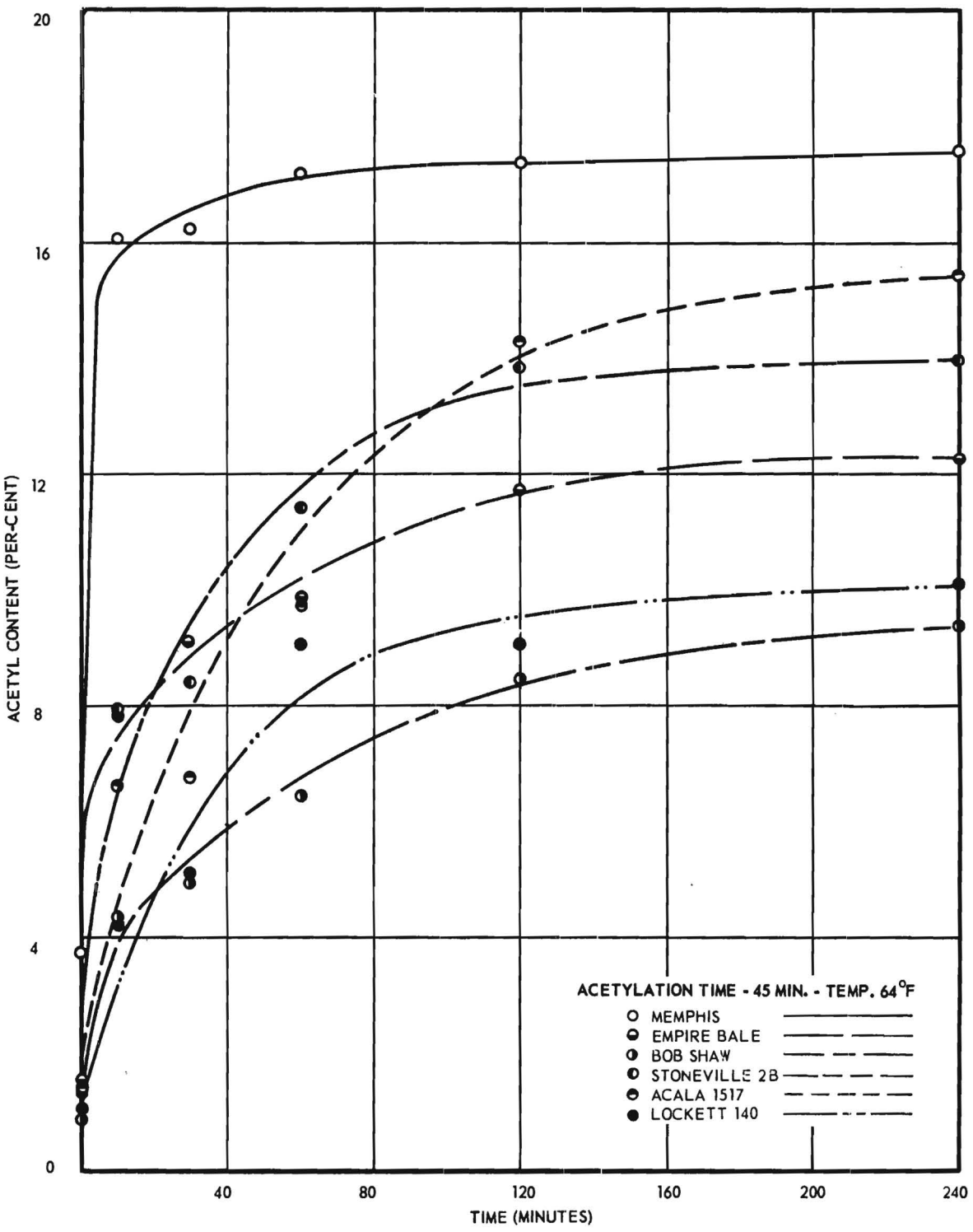

Figure 16. The Change in Acetyl Content with Presoaking Time at 100°F.

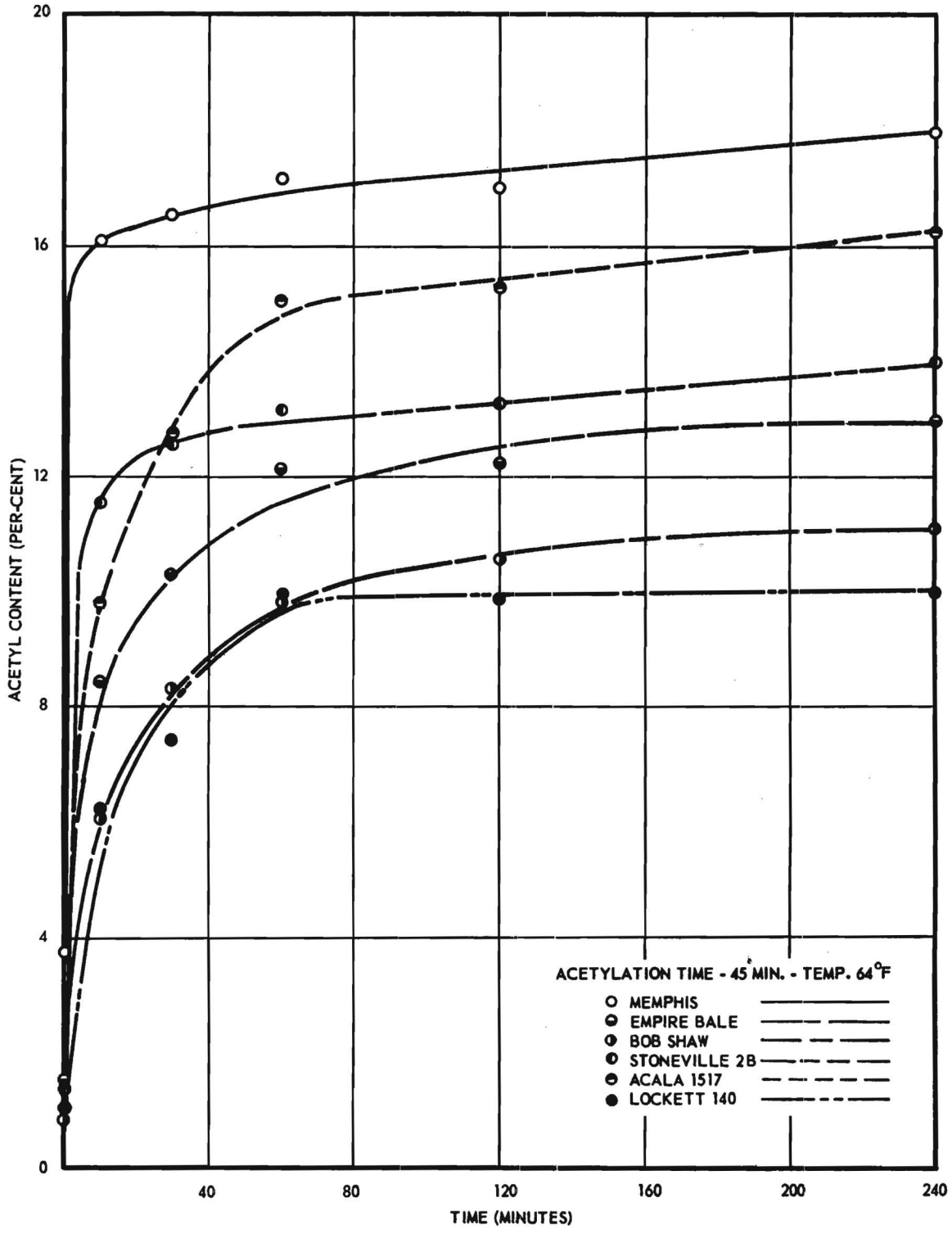

Figure 17. The Change in Acetyl Content with Presoaking Time at 130°F.

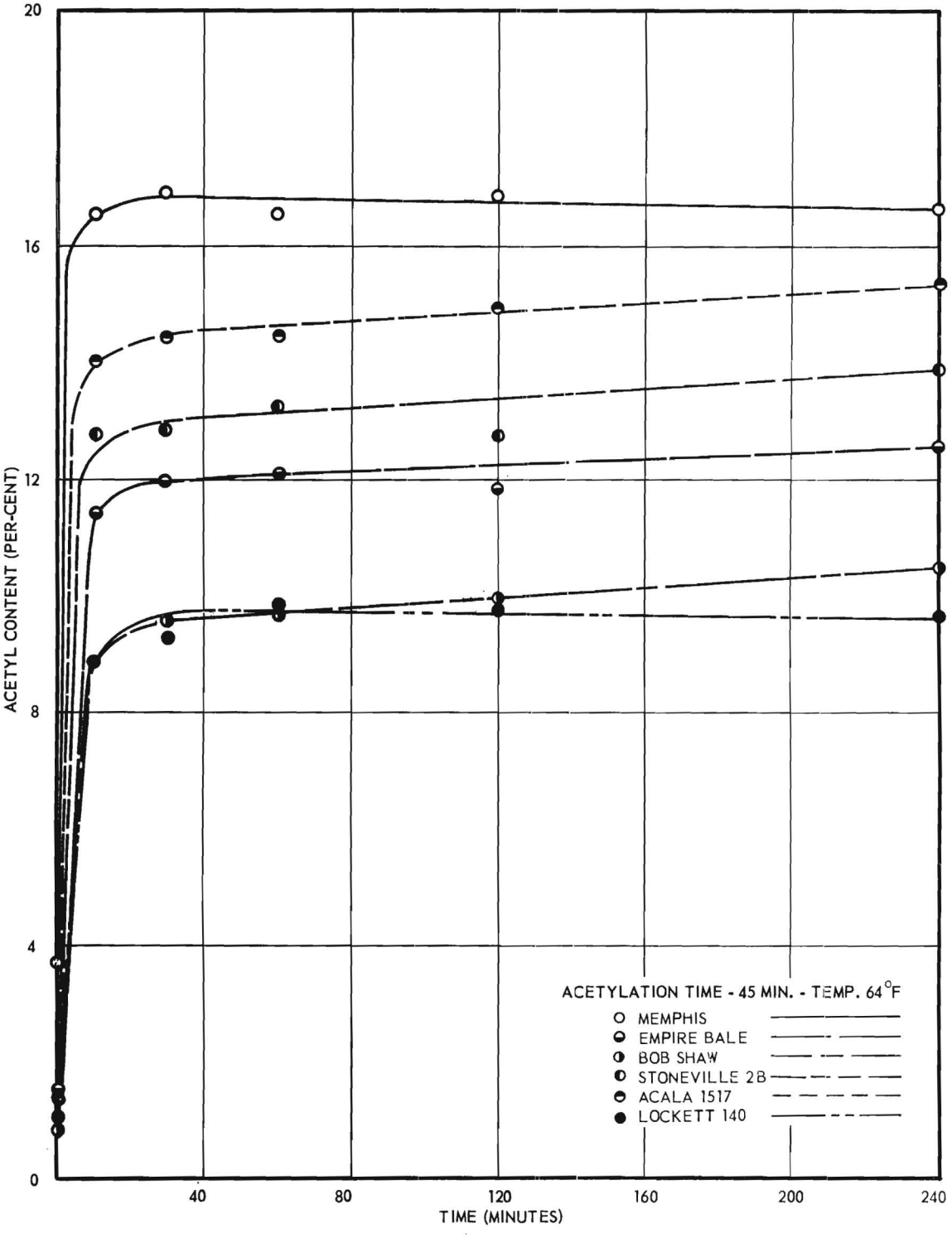

Figure 18. The Change in Acetyl Content with Presoaking Time at 170° F.

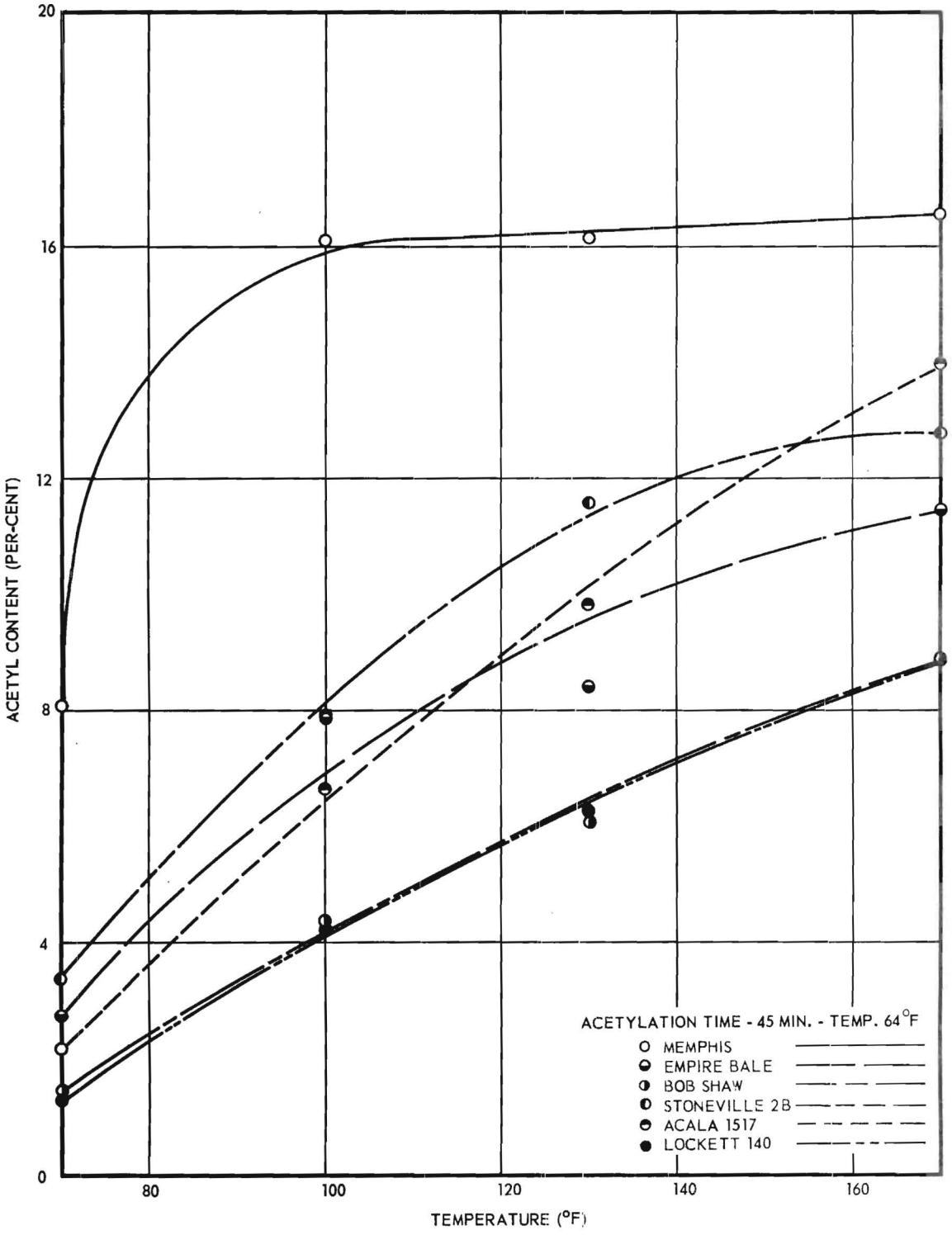

Figure 19. The Change in Acetyl Content with Presoaking Temperature Using a 10 Minute Presoaking Period.

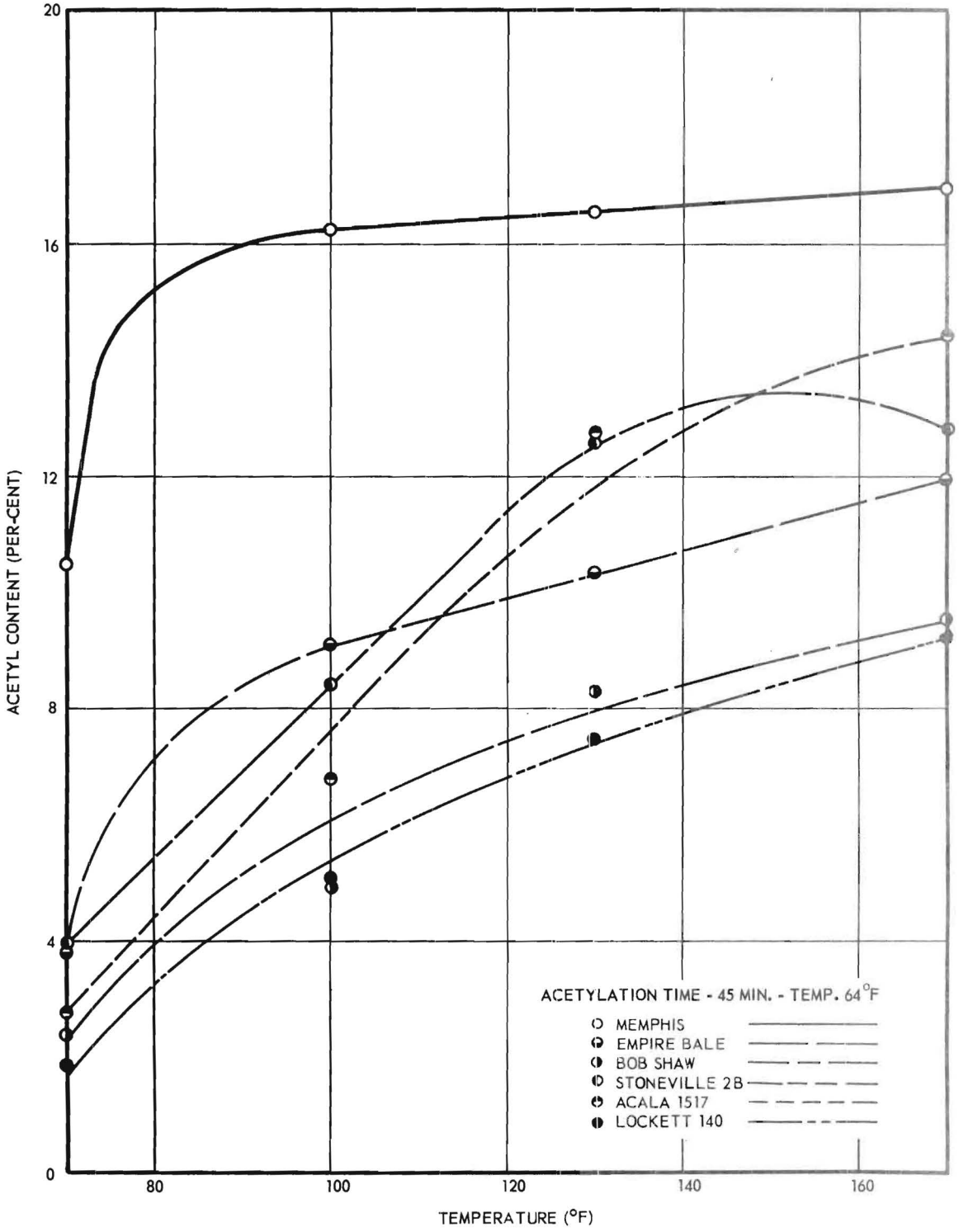

Figure 20. The Change in Acetyl Content with Presoaking Temperature Using a 30 Minute Presoaking Period.

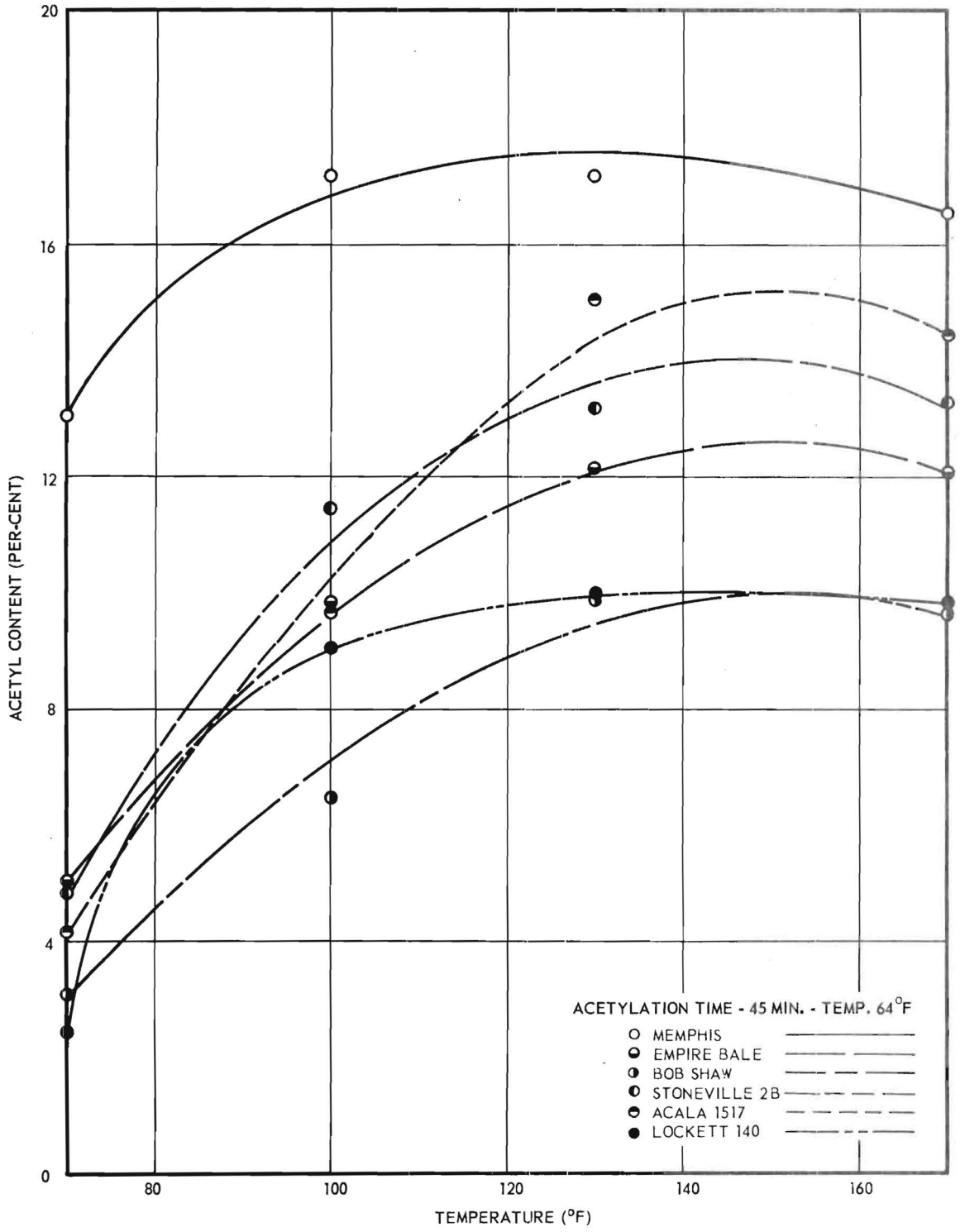

Figure 21. The Change in Acetyl Content with Presoaking Temperature Using a 60 Minute Presoaking Period.

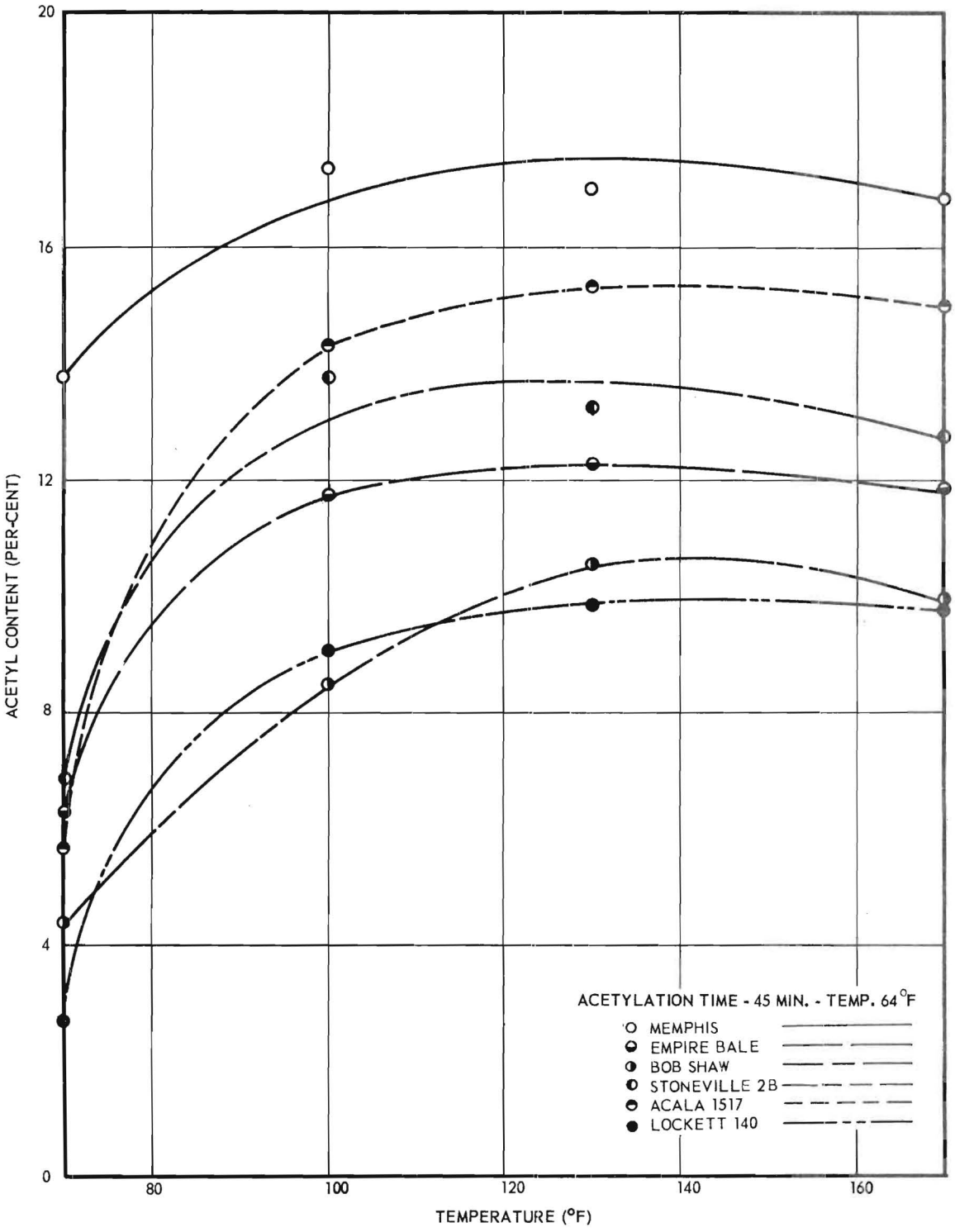

Figure 22. The Change in Acetyl Content with Presoaking Temperature Using a 120 Minute Presoaking Period.

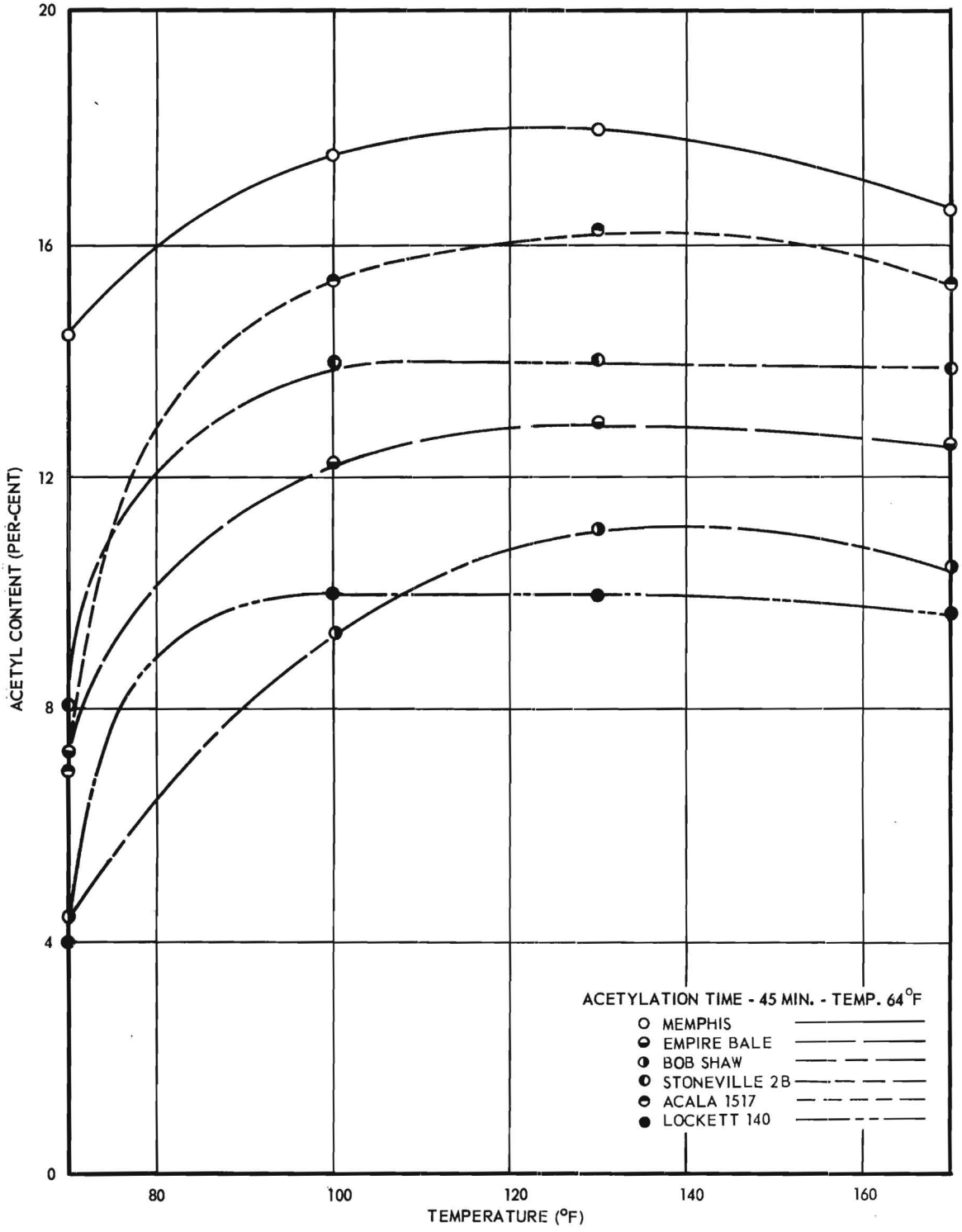

Figure 23. The Change in Acetyl Content with Presoaking Temperature Using a 240 Minute Presoaking Period.

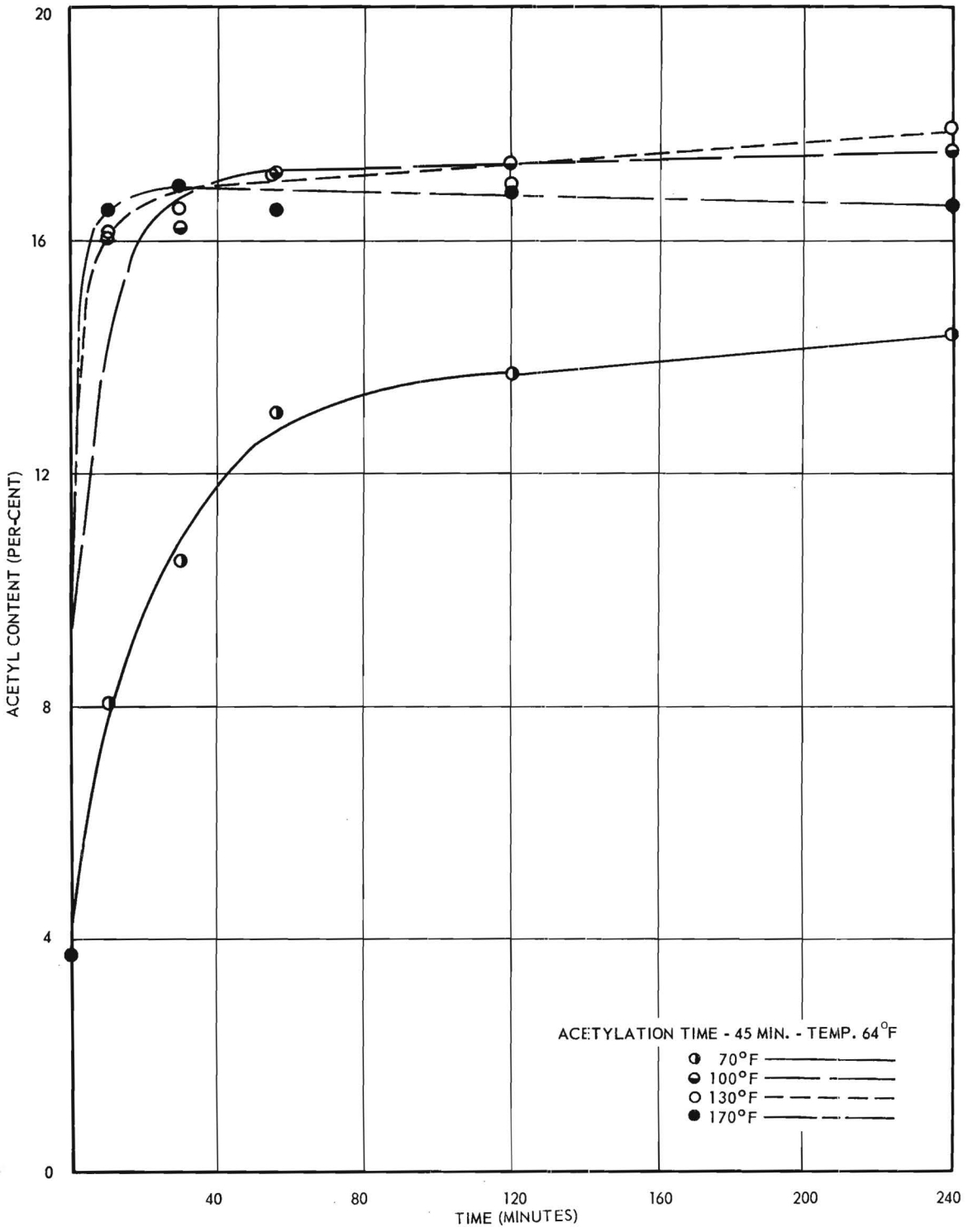

Figure 24. The Change in Acetyl Content with Presoaking Time for Memphis Cotton at Different Presoak Temperatures.

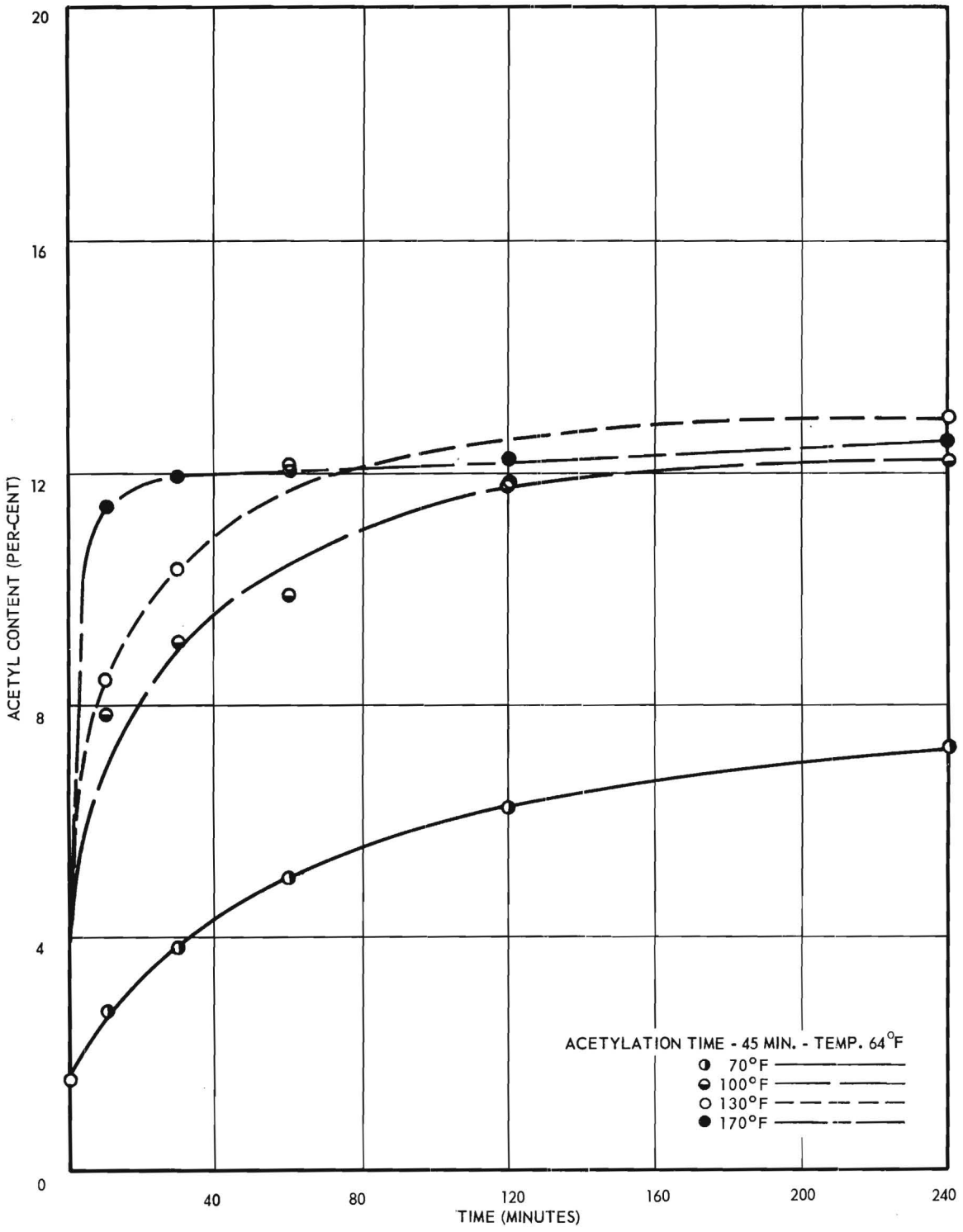

Figure 25. The Change in Acetyl Content with Presoaking Time for Empire Bale 92 Cotton at Different Presoak Temperatures.

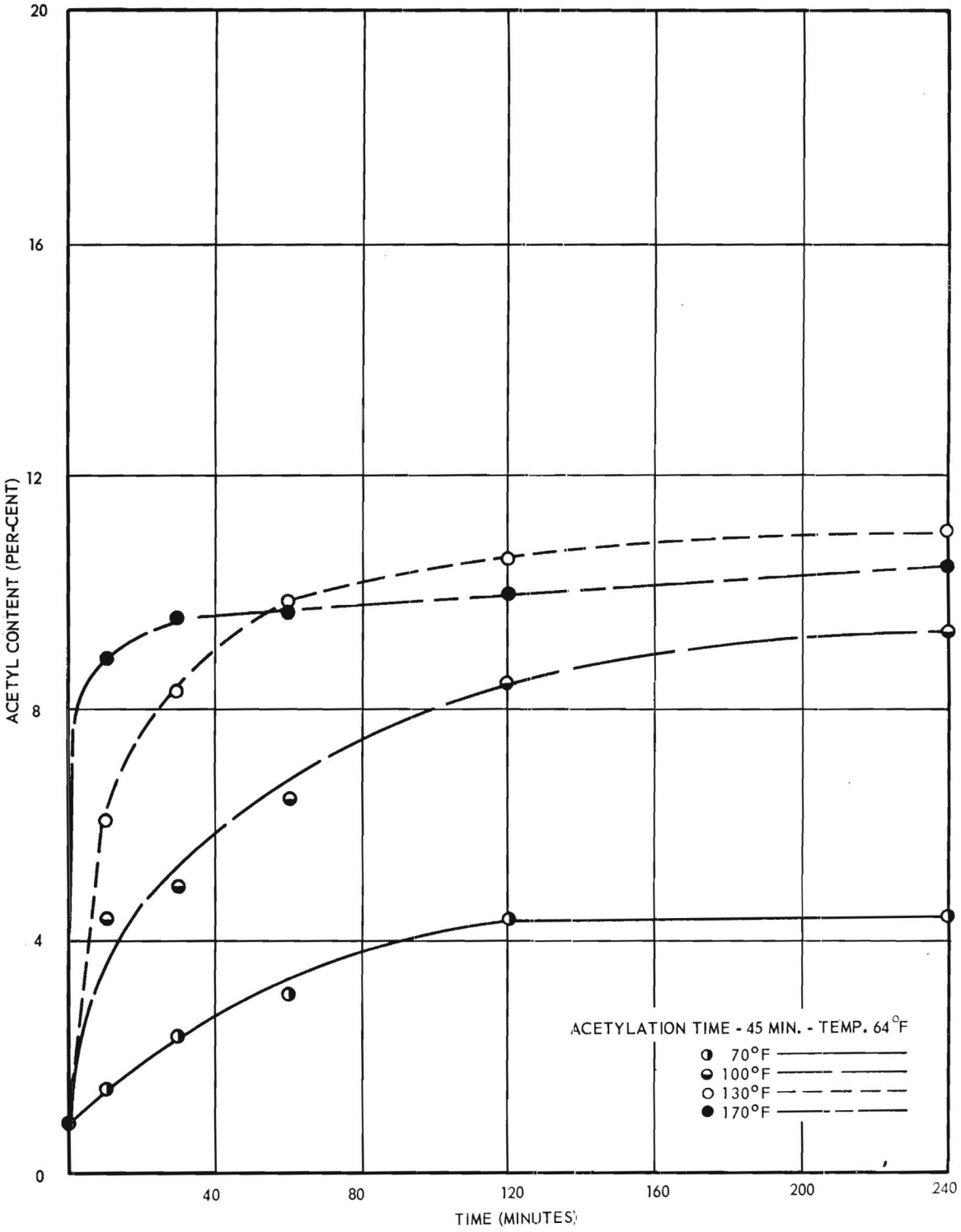

Figure 26. The Change in Acetyl Content with Presoaking Time for Bob Shaw Cotton at Different Presoak Temperatures.

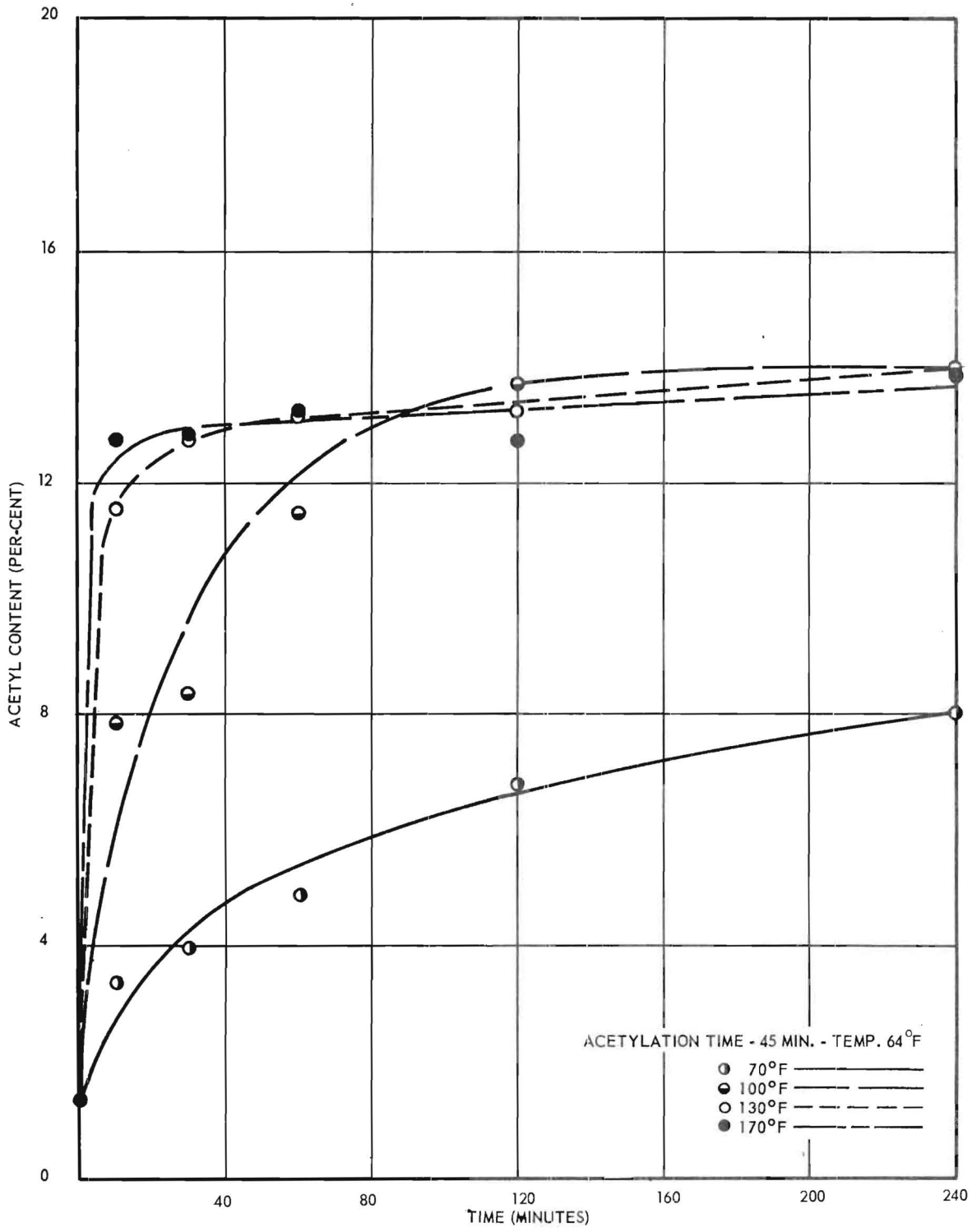

Figure 27. The Change in Acetyl Content with Presoaking Time for Stoneville 2B Cotton at Different Presoak Temperatures.

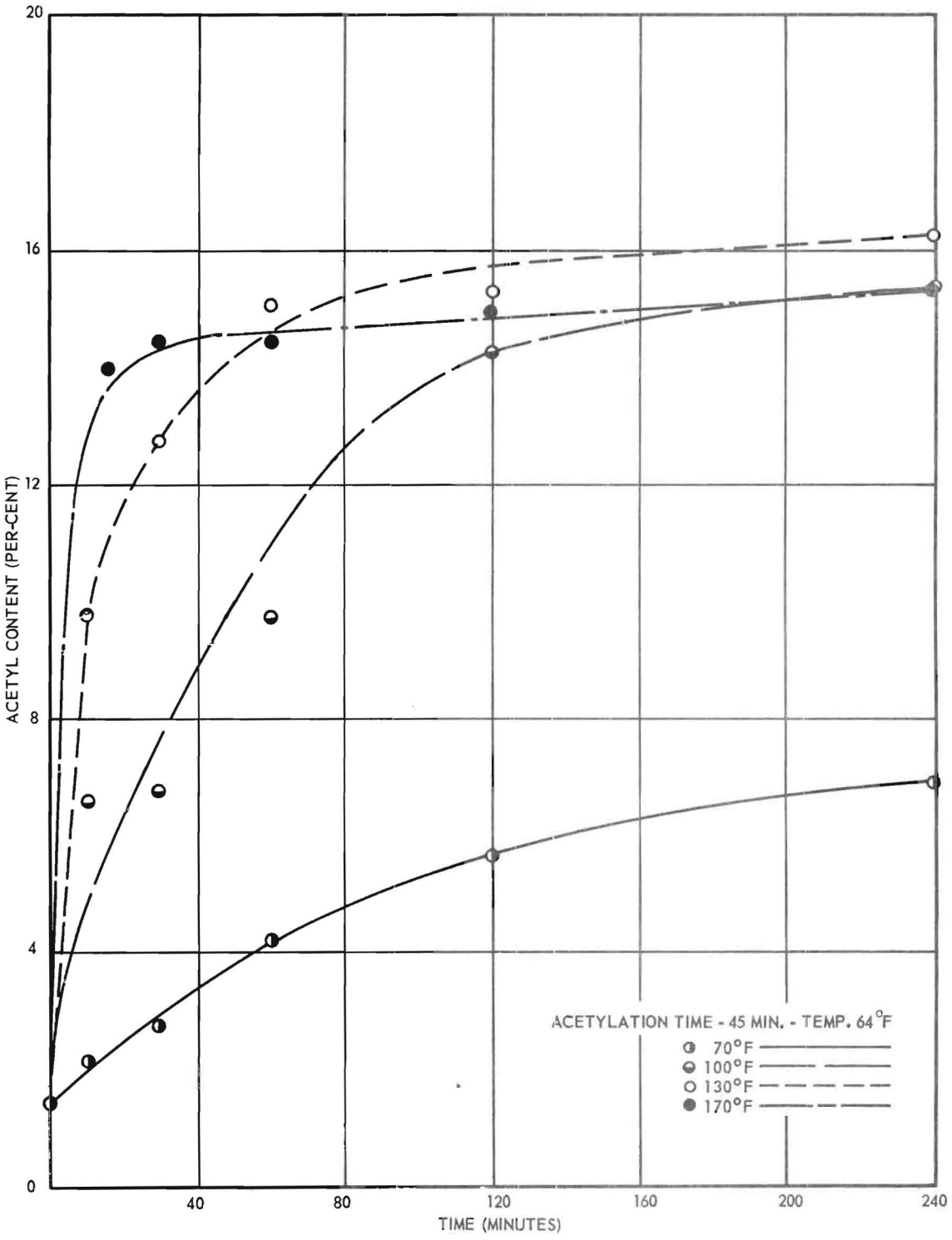

Figure 28. The Change in Acetyl Content with Presoaking Time for Acala 1517 Cotton at Different Presoak Temperatures.

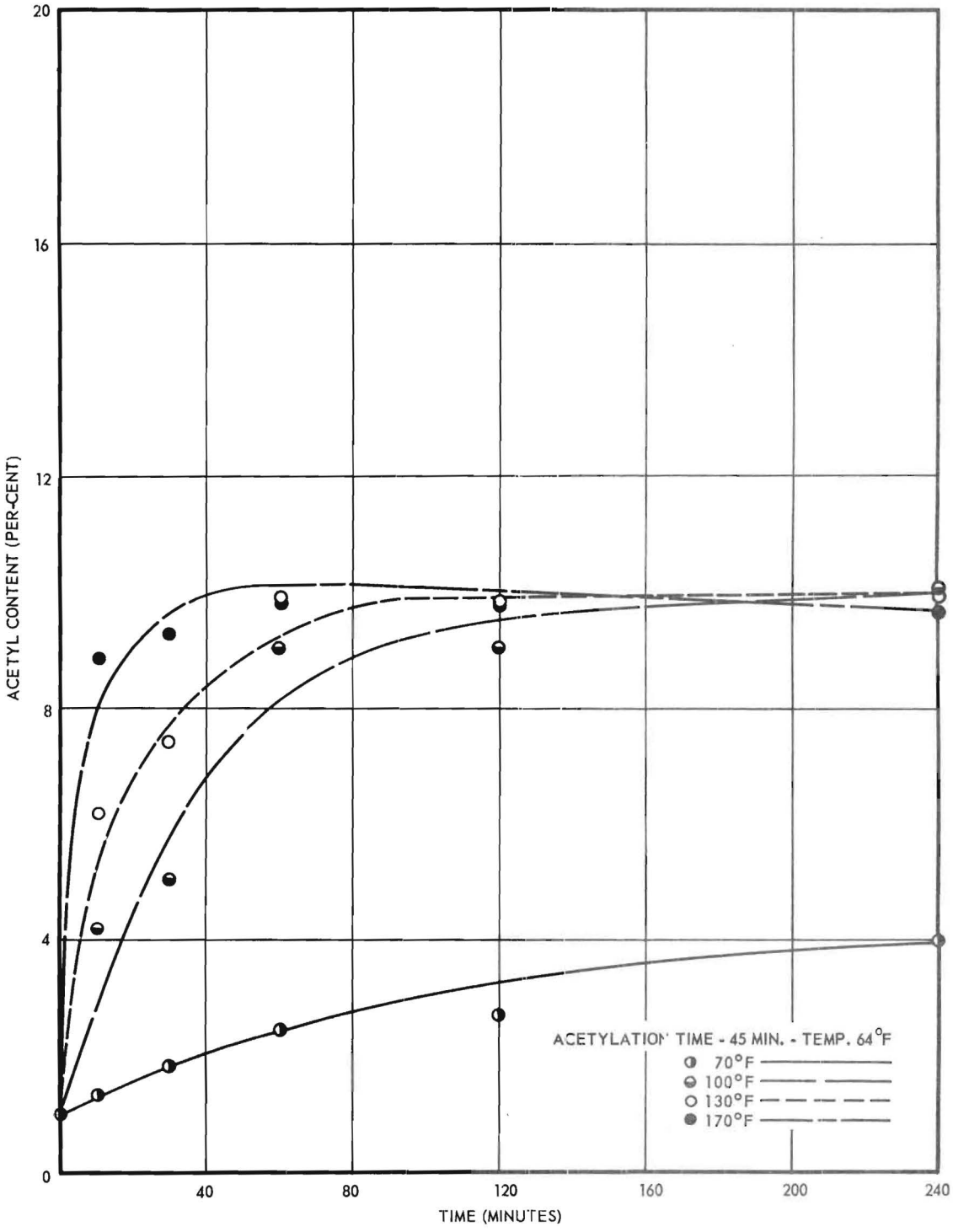

Figure 29. The Change in Acetyl Content with Presoaking Time for Lockett 140 Cotton at Different Presoak Temperatures.

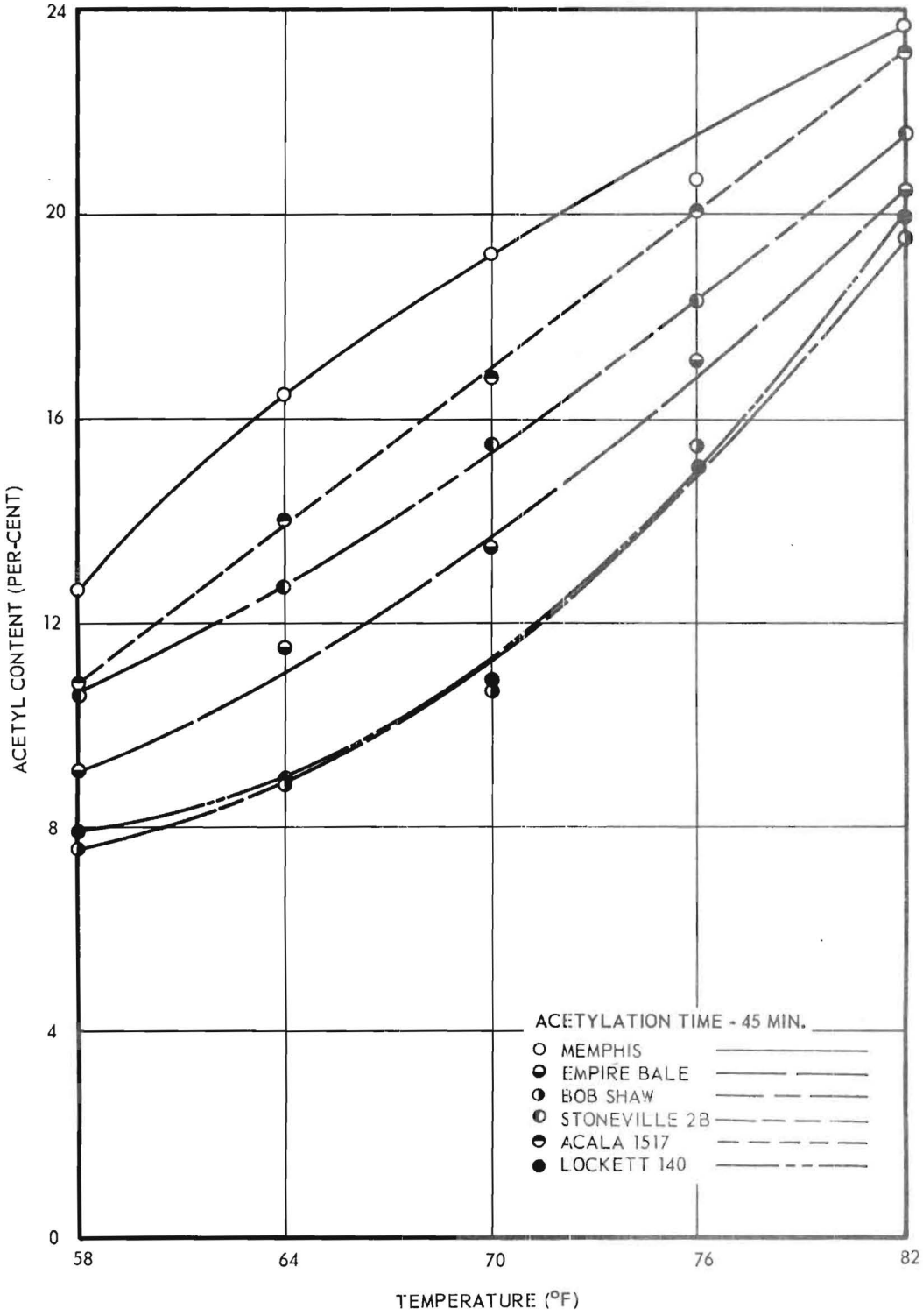

Figure 30. The Change in Acetyl Content with Acetylation Temperature Using a 10 Minute, 170° F. Presoak.

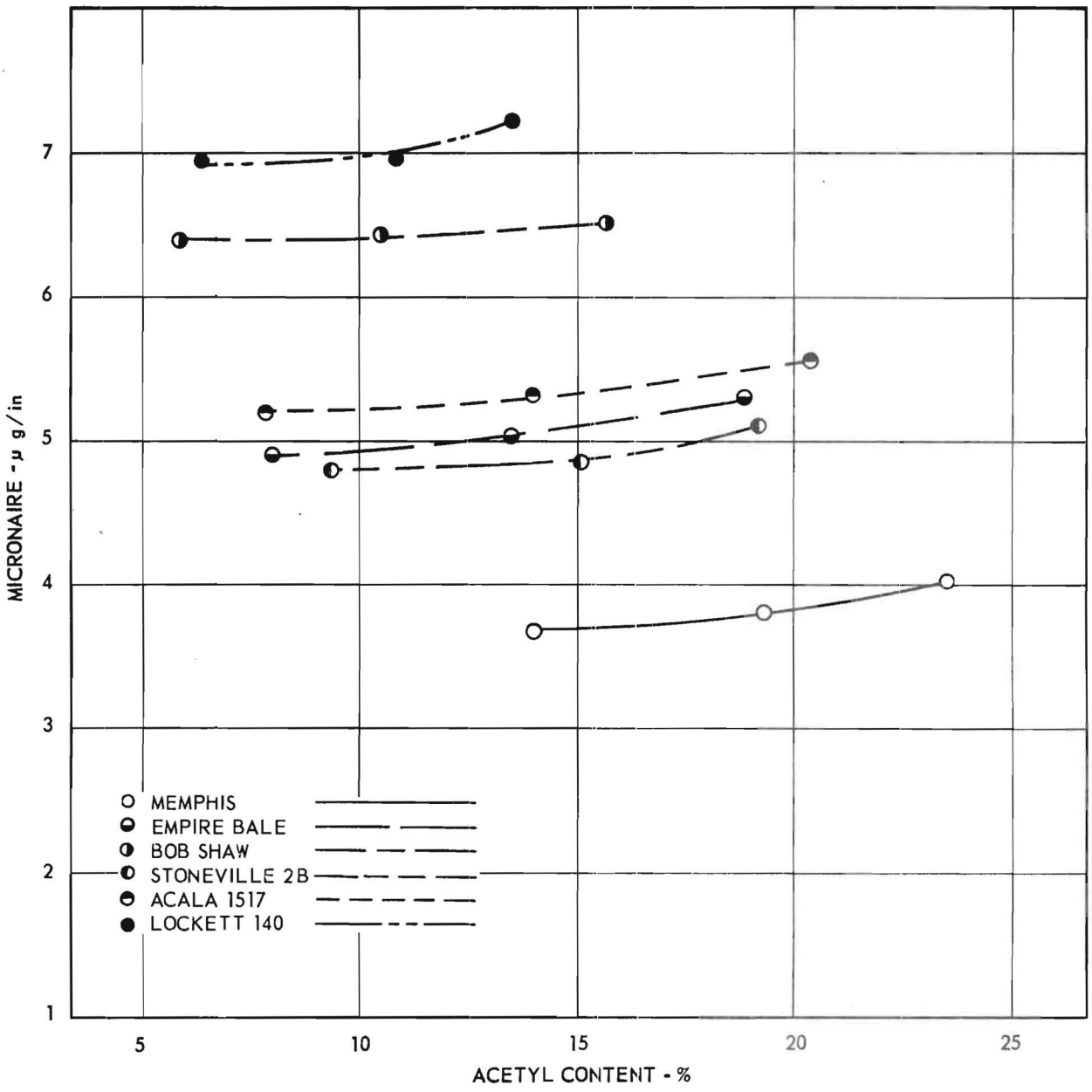

Figure 31. The Change Micronaire with Acetyl Content for Six Varieties of Cotton.

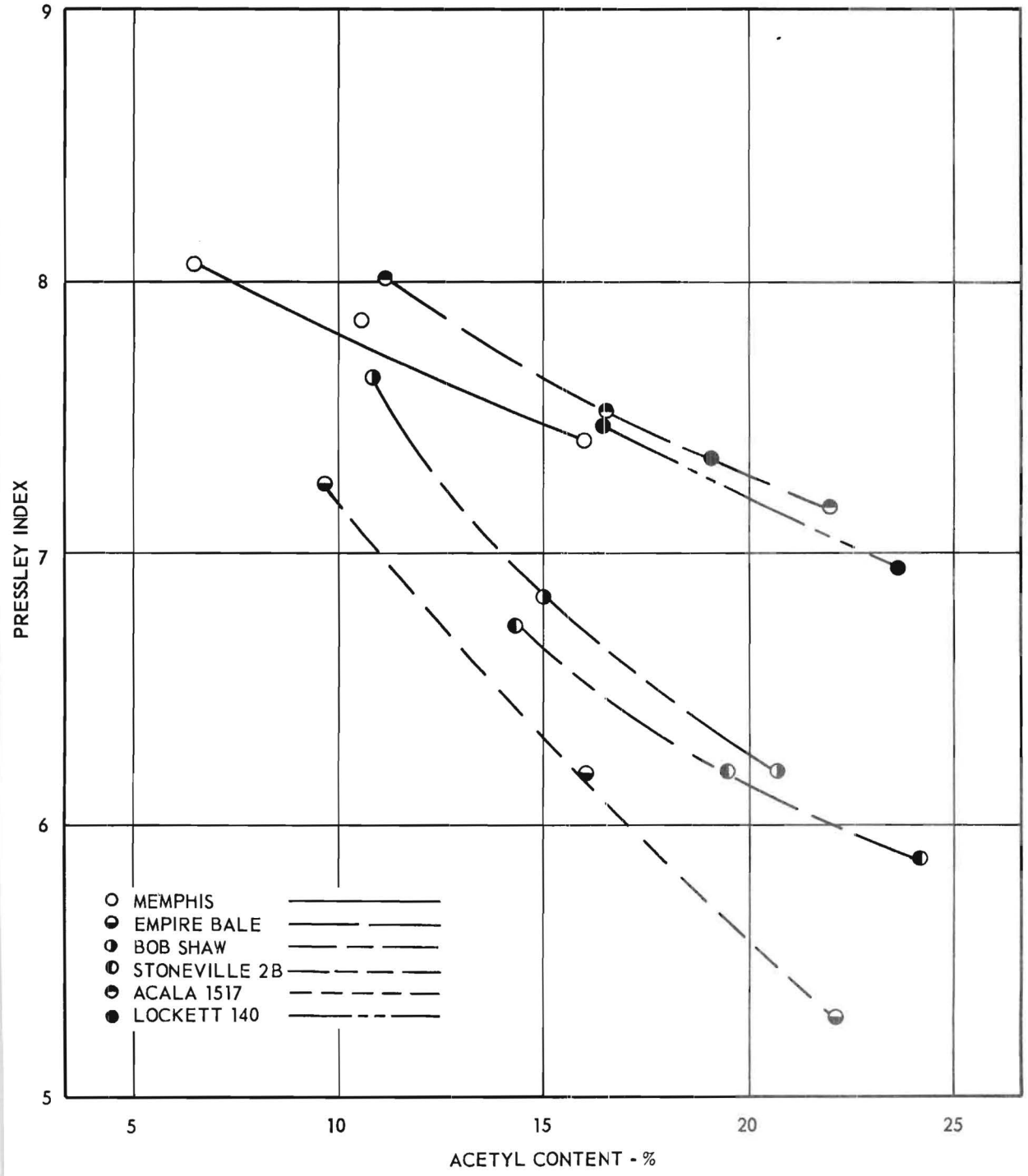

Figure 32. The Change in Pressley Index with Acetyl Content for Six Varieties of Cotton.

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 6

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

SEPTEMBER 20, 1953

UNITED STATES DEPARTMENT OF AGRICULTURE AGRICULTURAL RESEARCH ADMINISTRATION BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY	REPORT TO SOUTHERN REGIONAL RESEARCH LABORATORY NEW ORLEANS, LOUISIANA

CONTRACT PROJECT REPORT

CONTRACTING ORGANIZATION	PROJECT LEADERS	
Georgia Tech Research Institute	James L. Taylor	
DEPARTMENT	USDA DESIGNATED REPRESENTATIVE	
Research	Charles F. Goldthwait	
DIVISION	CONTRACT NUMBER	INITIATION DATE
Chemical Sciences	A-ls-33460	March 20, 1952
LOCALITY	REPORT NUMBER	PERIOD COVERED
Atlanta, Georgia	Six	June 20 - Sept. 20, 1953

TYPE OF REPORT: PROGRESS (X) PHASE ( ) ANNUAL ( ) TERMINATION ( )

PROJECT TITLE:

The Partial Acetylation of Cotton

ABSTRACT OF PROGRESS

During the past quarter 25 cottons, representing a wide range of properties, such as location of growth, variety, maturity, and fiber fineness, were acetylated at constant temperature and various acetylation times. The previously reported data for the acetylation of the first twelve cottons should be ignored since the acetylation temperature could not be kept constant throughout the reaction time. Therefore, when modifications in the acetylation machine permitted acetylating at constant temperature, these twelve cottons were again acetylated along with the thirteen others recently received from Southern Regional Research Laboratory. The resulting rate curves for the acetylation of each cotton are included in this report. It was determined that, in general, the acetylation rate for each cotton follows an exponential curve, and as a result an equation representing the acetylation reaction of each cotton can be written.

Determinations were made of the amount of ash in three varieties of unscoured and scoured cottons. These cottons were selected because of widely different acetylation rates, maturity, and fiber fineness values. The resultant ash from each cotton was spectrographically analyzed for its constituents. Examination of the results indicates that neither the amount of ash nor the ash composition greatly affects the rate of acetylation.

Preliminary statistical calculations have been made to determine the effects of the physical properties of the various cottons on their rates of acetylation. The results of these statistical studies will be presented next quarter when the complete data can be shown.

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 6

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

SEPTEMBER 20, 1953

TABLE OF CONTENTS

This Report Contains 21 Pages

LIST OF TABLES

LIST OF FIGURES

I. WORK PROGRAM

The work program followed for the past quarter is presented below.

1. The rates of acetylation at 64° F. have been determined for 25 different cottons during the past quarter.
2. Determinations of per cent ash and spectrographic analyses for the constituents of this ash have been made on scoured and unscoured samples of Memphis, Empire, and Lockett 140 cottons.
3. Attempts have been made to relate the rate of acetylation of each cotton with its physical properties. Statistical studies of the effects of the scouring, conditioning at various relative humidities, and presoaking experiments on the acetylation reaction have been started.

II. EXPERIMENTAL WORK

A. Acetylation of 25 Different Cottons

The twelve cottons (1 - 12, Table I) originally received from SRRL along with thirteen additional cottons were selected for determination of acetylation rates. It was thought advisable to repeat the acetylation experiments on the original twelve cottons since initial attempts to acetylate at constant temperature were unsatisfactory because constant temperatures could not be maintained. Subsequently, the acetylation equipment was modified to provide the control necessary to acetylate at constant temperature. (See Progress Report No. 4, page 2.)

The additional thirteen cottons were selected to show if differences in area of growth, per cent maturity, and fiber fineness affect the acetylation rates.

These 25 cottons, listed in Table I, were cleaned, conditioned at 70° F. and 65 per cent relative humidity, then presoaked in glacial acetic acid at 70° F. for approximately 18 hours prior to acetylation at 64° F. for 15, 30, 60, and 120 minutes using the procedure described in Progress Report No. 4, section B, page 3. These results are shown in Table II and Figures 1 through 6.*

B. Determination of Ash Content and Ash Analyses of Three Varieties of Cotton

Three varieties of cotton (Memphis, Lockett 140, and Empire) were selected for chemical analysis in order to determine the relationship between rate of acetylation and the chemical constituents of cotton such as: per cent ash, ash constituents, per cent water-soluble materials, acetic-acid-soluble materials, and others as specified in the contract.

These cottons, selected because of wide differences in maturity, fiber fineness, and rate of acetylation, were tested for per cent ash constituents.

For the determination of ash content an approximately 10 gram sample of each cotton was accurately weighed in a platinum crucible. The crucible and cotton were then placed in a muffle furnace and ashed at approximately 650° C. After cooling in a desiccator, the sample was weighed and the per cent ash calculated.

The ash from duplicate samples was then combined and the constituents determined by spectrographic analysis.

Scoured samples of the three cottons previously mentioned (scoured in a solution of 1.0 per cent Duponol RA and 1.5 per cent tetrasodium pyrophosphate for 30 minutes at the boil in a 20-to-1 bath) were also tested for per cent ash

* - - - -
*All Tables and Figures are presented in the appendix.

and ash constituents, using the same method outlined above for the unscoured samples. The results of all these tests are presented in Tables III and IV.

C. Statistical Studies of the Effects of the Physical Properties on the Acetylation of Different Cottons

Preliminary calculations to determine if there is any correlation between the physical properties and the rate of acetylation of the various cottons have been undertaken. Analysis of variance calculations for the scouring, relative humidity conditioning, and presoak experiments have also been undertaken. Since all statistical calculations have not been completed, no correlation data is included in this report.

III. DISCUSSION OF RESULTS

In addition to the original 12 cottons listed in Table I, Progress Report No. 2, 13 cottons which exhibit differences in growth, location, variety, maturity, fineness values, strength, and x-ray angle were acetylated in order to determine the effect of physical properties on rates of acetylation, as illustrated in Figures 1 through 6. These cottons show a wide range of acetylation rates.

Figures 7 through 10 show a plot of $\log \frac{A}{T}$ vs. $\log T$ where A is per cent acetyl and T is time. The fact that these plots are straight lines permits numeric representation of those curves for each cotton by the general equation:

$$\log \frac{A}{T} = \log K + N \log T$$

where A is per cent acetyl, T is acetylation time, K and N are constants for each cotton. This numerical characterization of the cottons will be useful in the correlation calculations which will be completed and reported next quarter.

Examination of the data from the ash determinations shows a slight increase in ash content for mature cottons. The analysis for the ash constituents as presented in Table II reveals little difference in the constituents of the samples. It is noted that the very immature Memphis cotton shows traces of aluminum and phosphorus in addition to other elements found in the more mature Empire and Lockett. Further examination shows that the scouring action removes all of the potassium, does not essentially change the amounts of sodium, magnesium, silicon, and boron, but increases the amounts of calcium and aluminum and adds some strontium. The addition of strontium and the increase in the amounts of aluminum and calcium is probably due to the scouring ingredients. An examination of the complete data, plus the effect of the scouring procedures as reported in Progress Report No. 4, indicates that neither the amount of ash nor the ash constituents greatly influences the rate and degree of acetylation.

IV. FUTURE PROGRAM

Using the data obtained during the past nine months, efforts will be directed toward determining the correlation of the physical properties of the various cottons with their rate of acetylation.

Statistical studies of the effect of scouring, relative humidity, conditioning, and presoaking on the rate of acetylation of cotton will also be made.

Various chemical analyses such as determination of per cent acetic-acid-, ammonium-oxalate-, ethyl-alcohol-, chloroform-, hot-water-, and benzene-soluble materials present in unscoured and scoured samples of Memphis, Empire, and Lockett 140 cottons will be completed next quarter.

Additional acetylations will be made on the six cottons used in the relative humidity study. Acetylations will be made at 30, 60, and 90 minutes on cotton conditioned at 0 per cent relative humidity and 100 per cent relative humidity in order to complete this study.

Respectfully submitted:

James L. Taylor
Project Director

Approved:

Herschel H. Cudd, Director
Engineering Experiment Station

V. APPENDIX

TABLE I

PHYSICAL PROPERTIES OF THE COTTONS
SELECTED FOR ACETYLATION

Cotton	Source	Maturity (NaOH)	Fineness (μgm/in)	Pressley Index	X-Ray Angle (40%)
1. Indian J & J	--	93	7.3	4.85	34.98
2. Sea Island	--	86	2.4	8.66	31.36
3. Memphis	--	38	2.5*	7.15*	37.92
4. Empire Bale 92	--	72	3.7*	7.30*	34.05
5. Stoneville 2B Bale 616654	--	79	3.9	7.4	---
6. Bob Shaw	--	88**	5.1*	8.17*	31.32
7. Stoneville 2B Bale 249290	--	80	3.2*	8.07*	31.20
8. Acala 1517	--	86	4.0*	8.93*	29.76
9. S x P Bale 3109	--	86	8.9	8.30	---
10. Lockett 140	--	92	5.6*	7.51*	36.18
11. Pima 32	--	84	2.8	10.5	29.28
12. Hopi Acala 50	--	95	4.6	8.60	33.75
13. Acala 4-42	Greenville, Texas	77	3.9	9.24	28.8
14. Acala 5675	Florence, S. C.	81-82	4.2	8.22	32.6
15. Acala 1517W	Florence, S. C.	85	4.0	8.21	28.0

(Continued)

TABLE I (Continued)

PHYSICAL PROPERTIES OF THE COTTONS
SELECTED FOR ACETYLATION

Cotton	Source	Maturity (NaOH)	Fineness (μgm/in)	Pressley Index	X-Ray Angle (40%)
16. Coker 100W	Weslaco, Texas	77	4.5	6.96	33.6
17. Coker 100W	Florence, S. C.	86	4.6	5.91	35.5
18. Rowden 41B	College Station, Texas	81	4.8	7.75	29.6
19. Rowden 41B	Greenville, Texas	86-87	5.5	7.85	31.4
20. Rowden 41B	Florence, S. C.	85	5.8	6.84	35.9
21. S x P Bale 3110	--	93	3.8	8.7	--
22. Sea Island St. Vincent	--	80	1.8	10.1	--
23. Seabury Sea Island	--	75	3.1	---	33.0
24. Sealand 1 47-295	Florence, S. C.	81-82	3.4	7.71	31.6
25. Deltapine 14-43-35	College Station, Texas	72	3.4	8.18	30.8

*Measurements made at the Georgia Tech Textile Laboratory; all other data furnished by Southern Regional Research Laboratory.

**Maturity determined by arealometer.

TABLE II

RESULTS OF ACETYLATING COTTONS AT 64° F.
FOR VARIOUS TIMES

	Cotton	Per Cent Acetyl				
		15 Minutes	30 Minutes	60 Minutes	90 Minutes	120 Minutes
1.	Indian J & J	8.57	10.6	17.0	21.8	23.8
2.	Sea Island	5.30	8.02	15.5	21.3	23.0
3.	Memphis	10.1	14.1	20.2	23.9	25.5
4.	Empire Bale 92	5.28	8.48	15.4	18.5	22.6
5.	Stoneville 2B Bale 616654	4.30	7.33	12.9	17.2	20.7
6.	Bob Shaw	3.79	6.84	13.1	16.4	20.4
7.	Stoneville 2B Bale 249290	7.05	10.5	17.0	20.6	23.7
8.	Acala 1517	7.51	11.5	18.0	22.0	25.4
9.	S x P Bale 3109	3.21	6.49	11.8	15.7	19.8
10.	Lockett 140	3.91	6.65	12.0	16.5	20.9
11.	Pima 32	5.12	9.98	16.5	21.0	25.0
12.	Hopi Acala 50	6.32	9.95	16.9	21.1	25.3
13.	Acala 4-42 (Greenville, Texas)	4.66	8.30	14.2	19.6	23.2
14.	Acala 5675 (Florence, S. C.)	5.86	10.4	16.4	21.4	25.1
15.	Acala 1517W (Florence, S. C.)	5.78	10.6	16.7	22.6	24.8

(Continued)

TABLE II (Continued)

RESULTS OF ACETYLATING COTTONS AT 64° F.
FOR VARIOUS TIMES

Cotton	Per Cent Acetyl				
	15 Minutes	30 Minutes	60 Minutes	90 Minutes	120 Minutes
16. Coker 100W (Weslaco, Texas)	5.08	8.83	14.5	20.6	24.4
17. Coker 100W (Florence, S. C.)	4.97	8.88	13.9	19.1	22.8
18. Rowden 41B (College Station, Texas)	4.47	8.04	12.5	18.3	21.7
19. Rowden 41B (Greenville, Texas)	6.67	11.8	17.3	21.9	25.9
20. Rowden 41B (Florence, S. C.)	5.95	10.9	16.9	20.7	24.8
21. S x P Bale 3110	4.43	8.38	17.3	20.2	21.4
22. Sea Island St. Vincent	8.03	12.5	21.4	24.2	27.5
23. Seabury Sea Island	6.20	10.2	18.6	20.8	24.8
24. Sealand 1 47-295	7.11	12.4	20.4	24.8	28.4
25. Deltapine 14-43-35	4.92	9.66	15.4	21.6	25.5

TABLE III

ASH ANALYSIS FOR THREE VARIETIES OF COTTON

	Per Cent Ash					
	Unscoured			Scoured		
Run No.	Memphis	Empire Bale 92	Lockett 140	Memphis	Empire Bale 92	Lockett 140
Run 1	0.87	0.90	0.96	0.24	0.35	0.37
Run 2	0.86	0.90	0.96	0.26	0.37	0.37
Average	0.87	0.90	0.96	0.25	0.36	0.37

TABLE IV

SPECTROGRAPHIC ANALYSIS FOR COTTON ASH CONSTITUENTS

	Unscoured			Scoured		
Element	Memphis	Empire Bale 92	Lockett 140	Memphis	Empire Bale 92	Lockett 140
Calcium	*f	f	f	s	s	s
Strontium	--	--	--	f	f	f
Aluminum	t	--	--	w	w	w
Potassium	f	w	f+	--	--	--
Sodium	f+	f	f	f	f	f
Magnesium	f-	f+	f-	f	f-	f+
Silicon	w-	w	w-	w	w	w
Boron	w	w-	w	w-	t	w-
Phosphorus	t		--	--	--	--

*s = strong; f = fair; w = weak; t = trace; -- = not found.

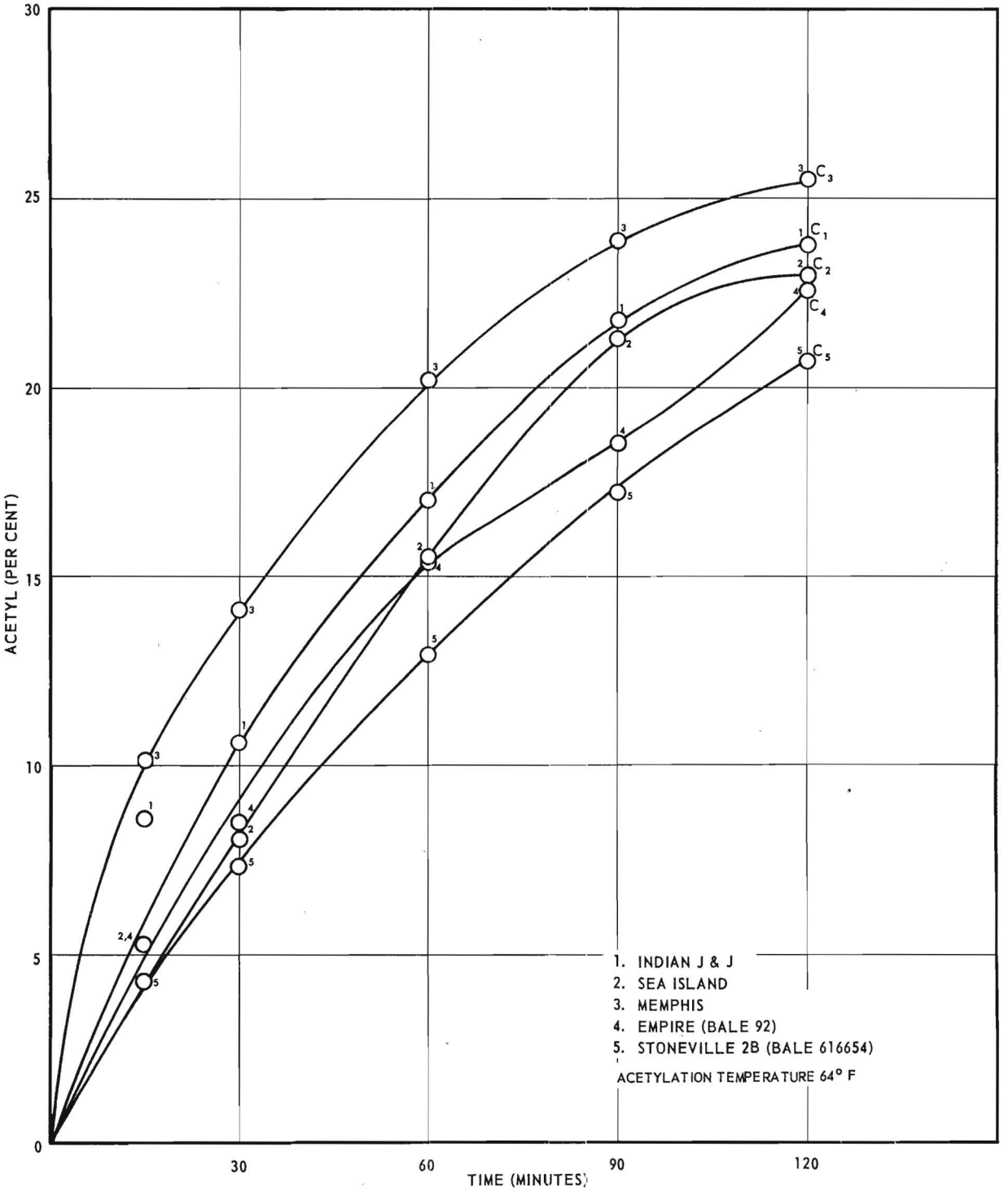

Figure 1. The Change in Acetyl Content with Time of Acetylation.

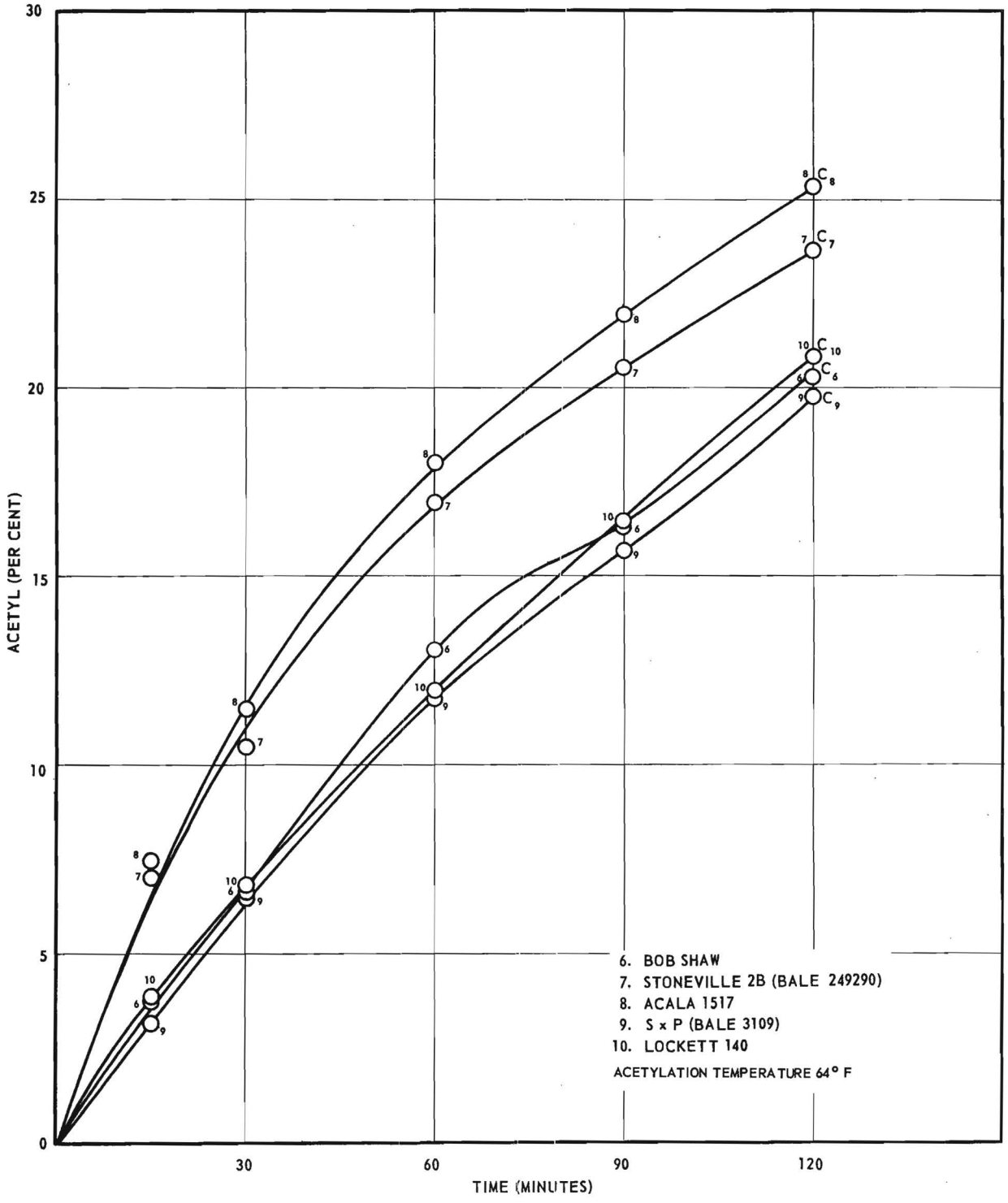

Figure 2. The Change in Acetyl Content with Time of Acetylation.

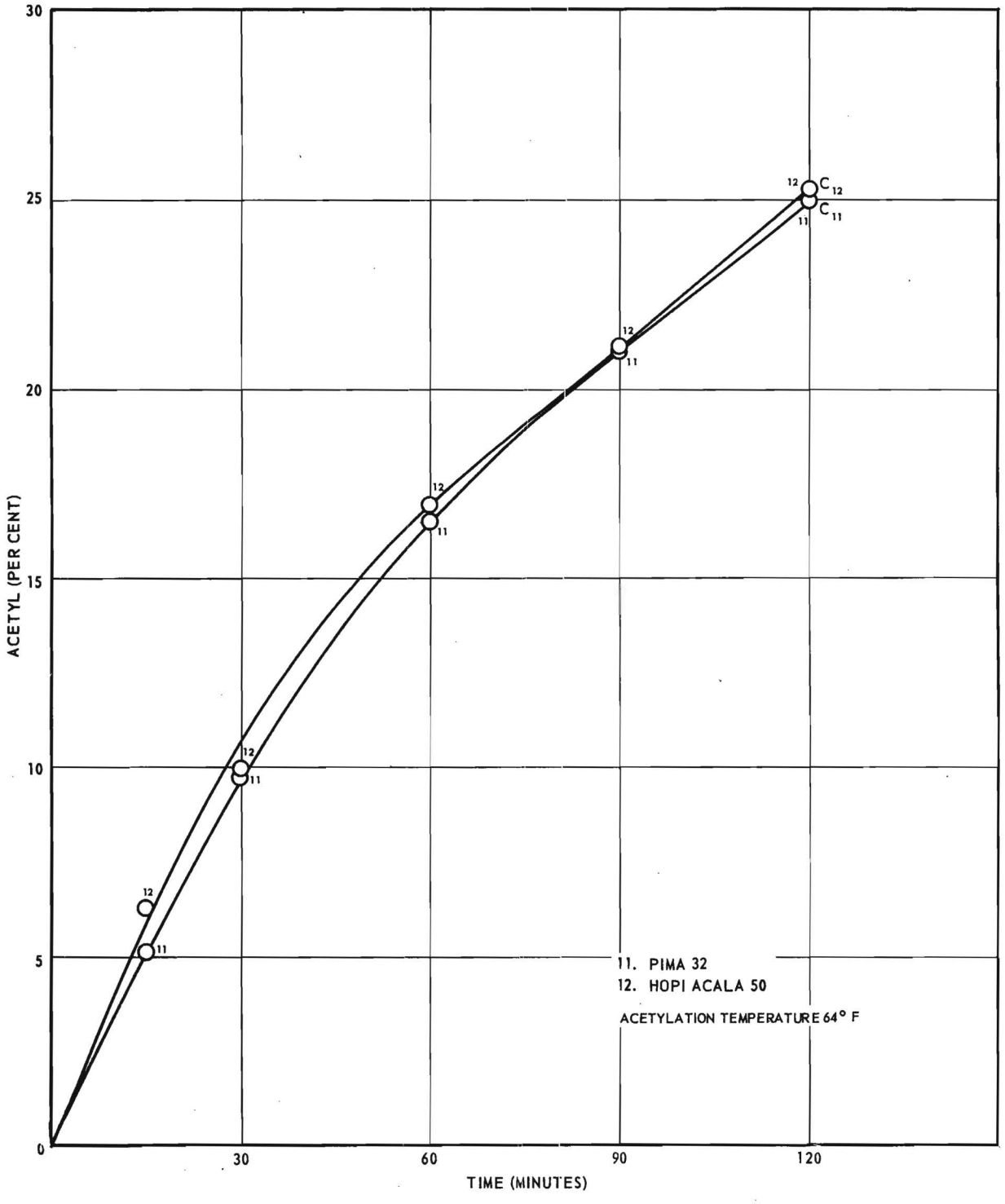

Figure 3. The Change in Acetyl Content with Time of Acetylation.

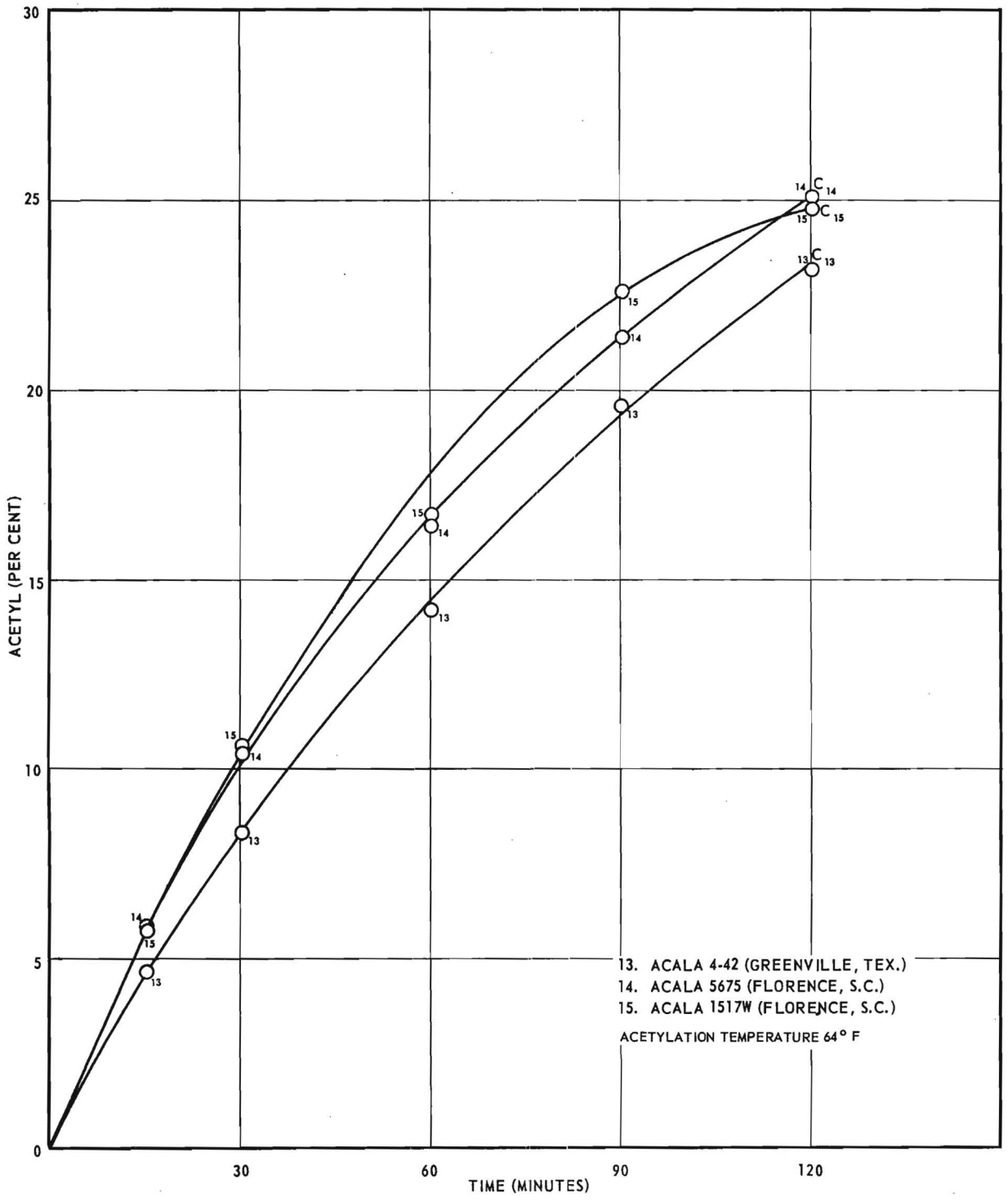

Figure 4. The Change in Acetyl Content with Time of Acetylation.

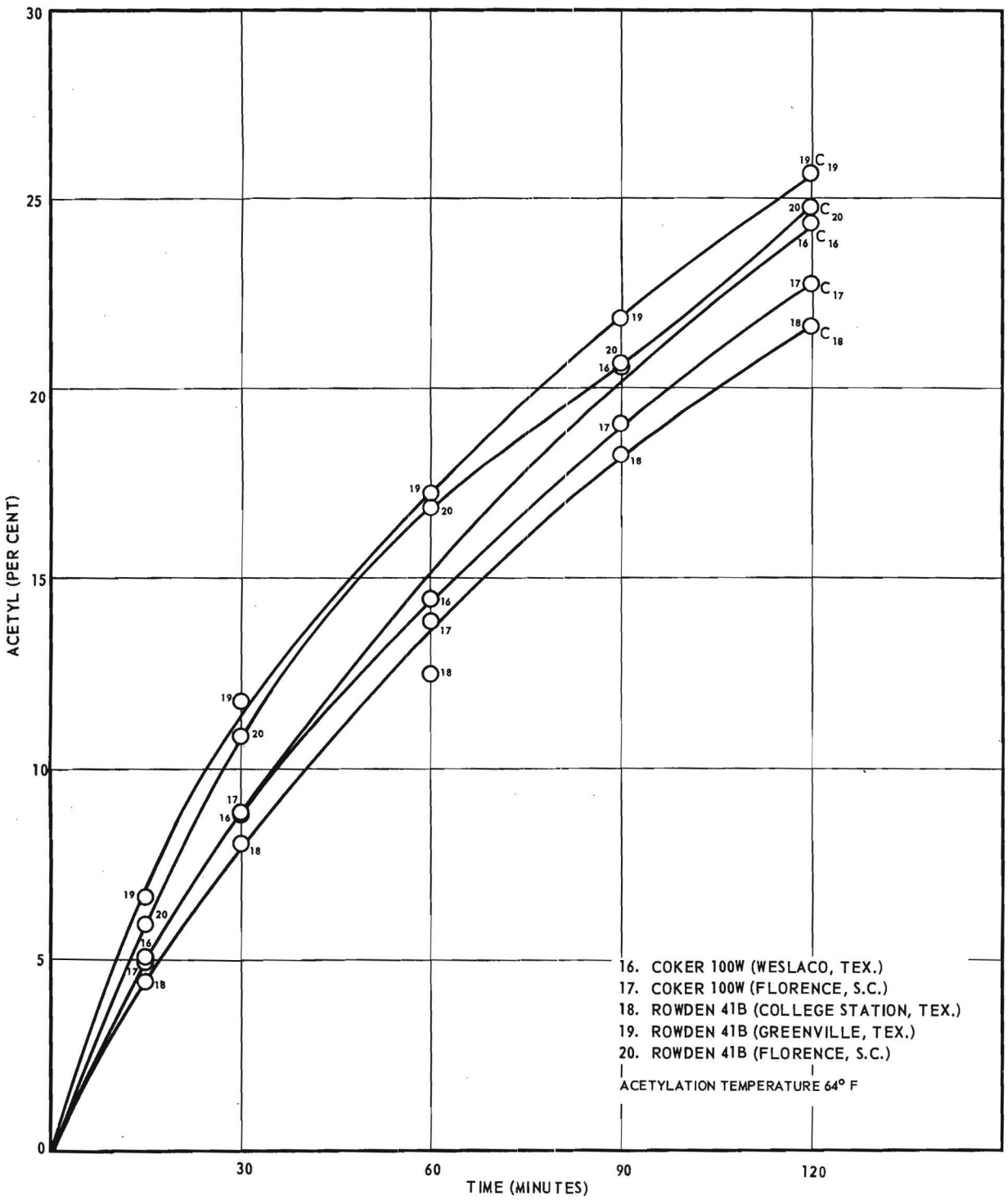

Figure 5. The Change in Acetyl Content with Time of Acetylation.

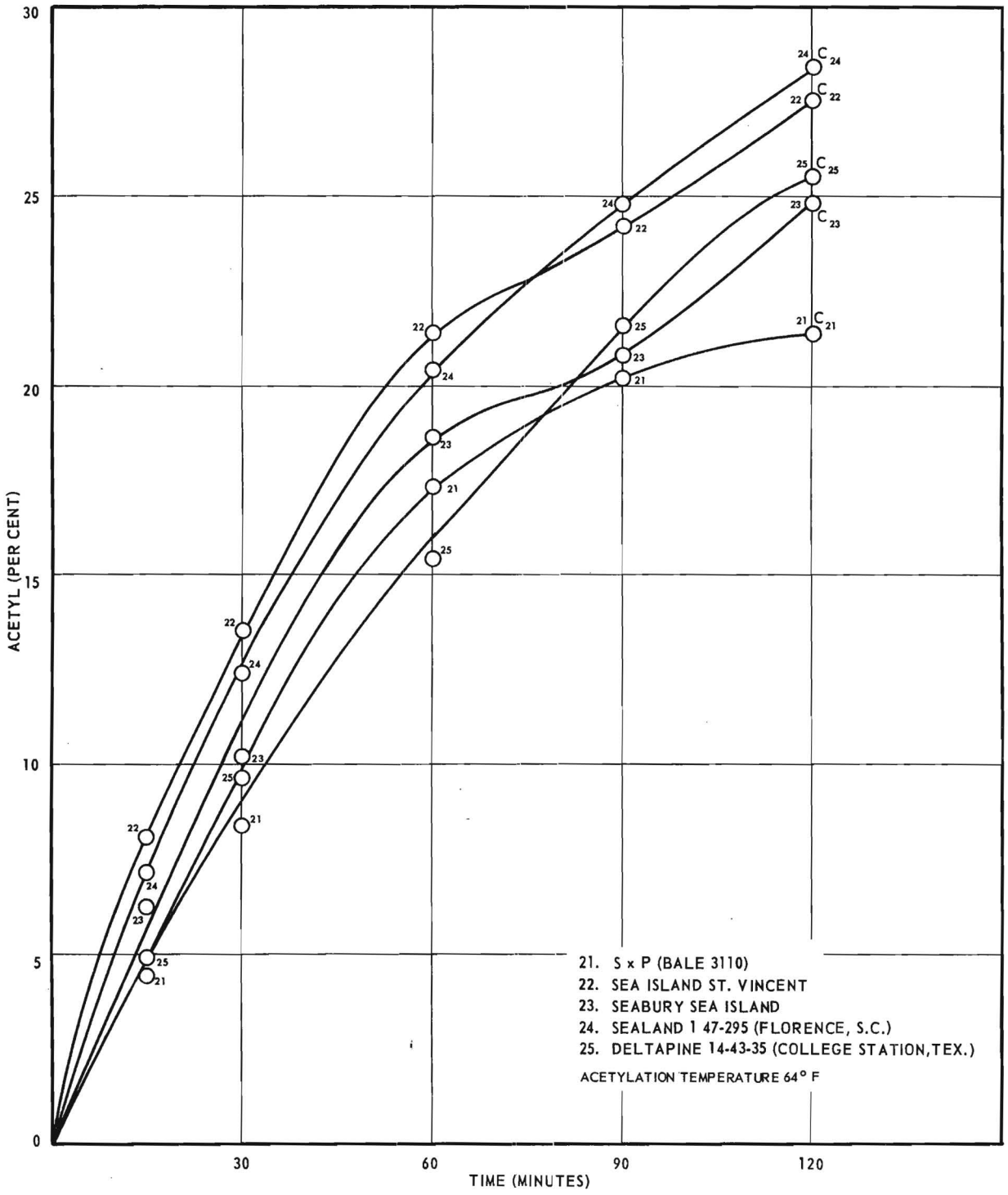

Figure 6. The Change in Acetyl Content with Time of Acetylation.

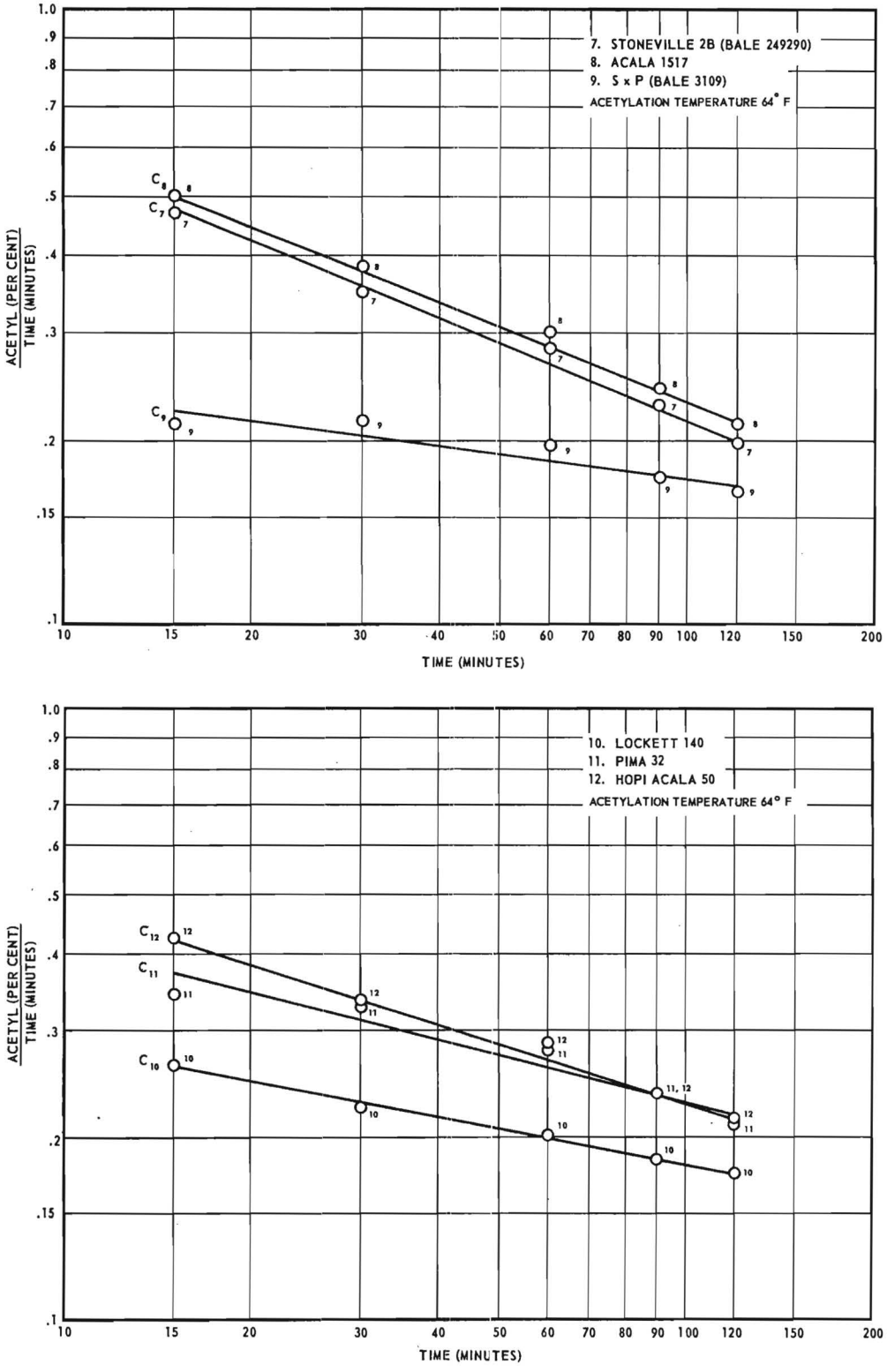

Figure 7. The Change in Rate of Acetylation with Time.

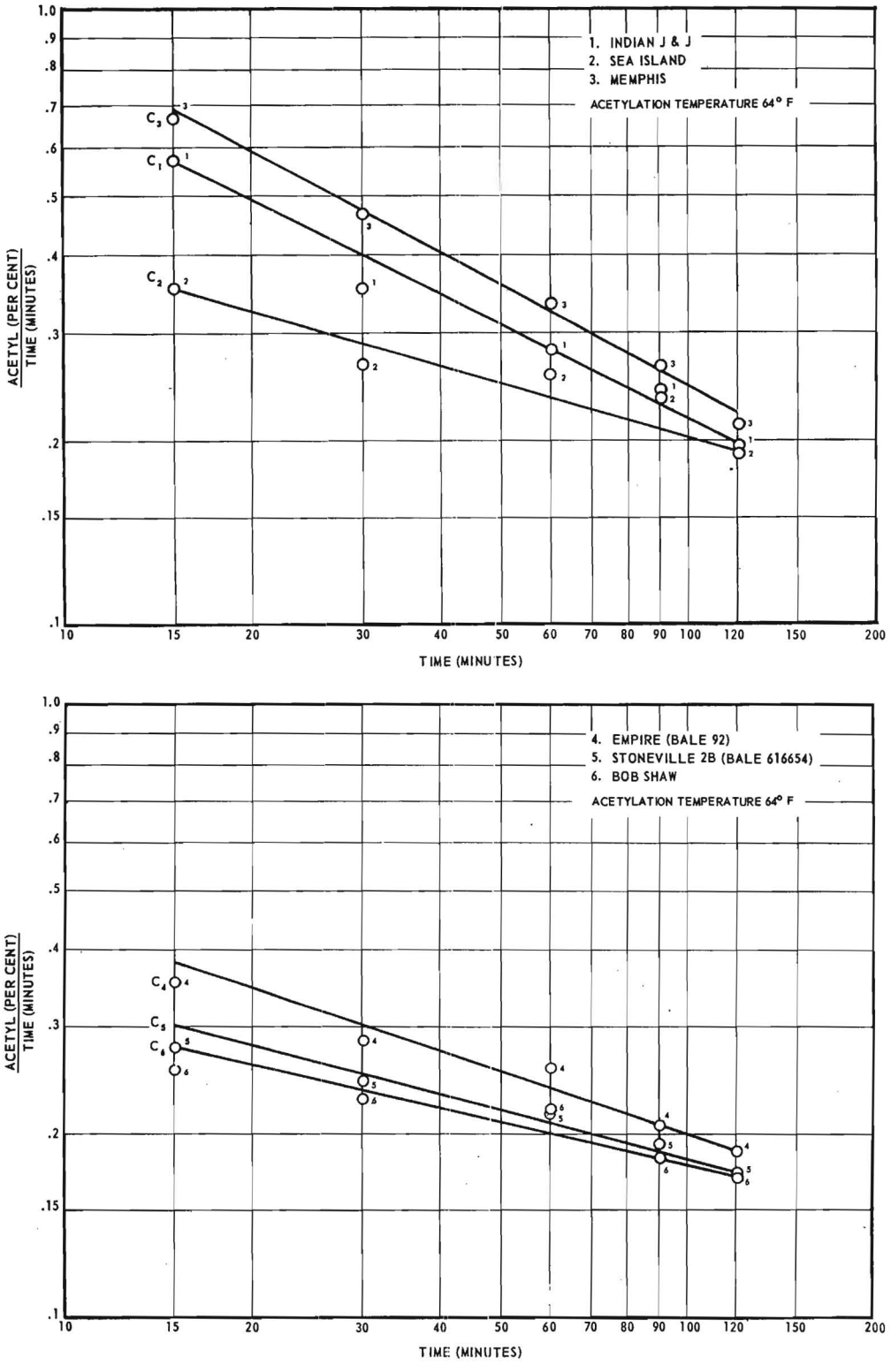

Figure 8. The Change in Rate of Acetylation with Time.

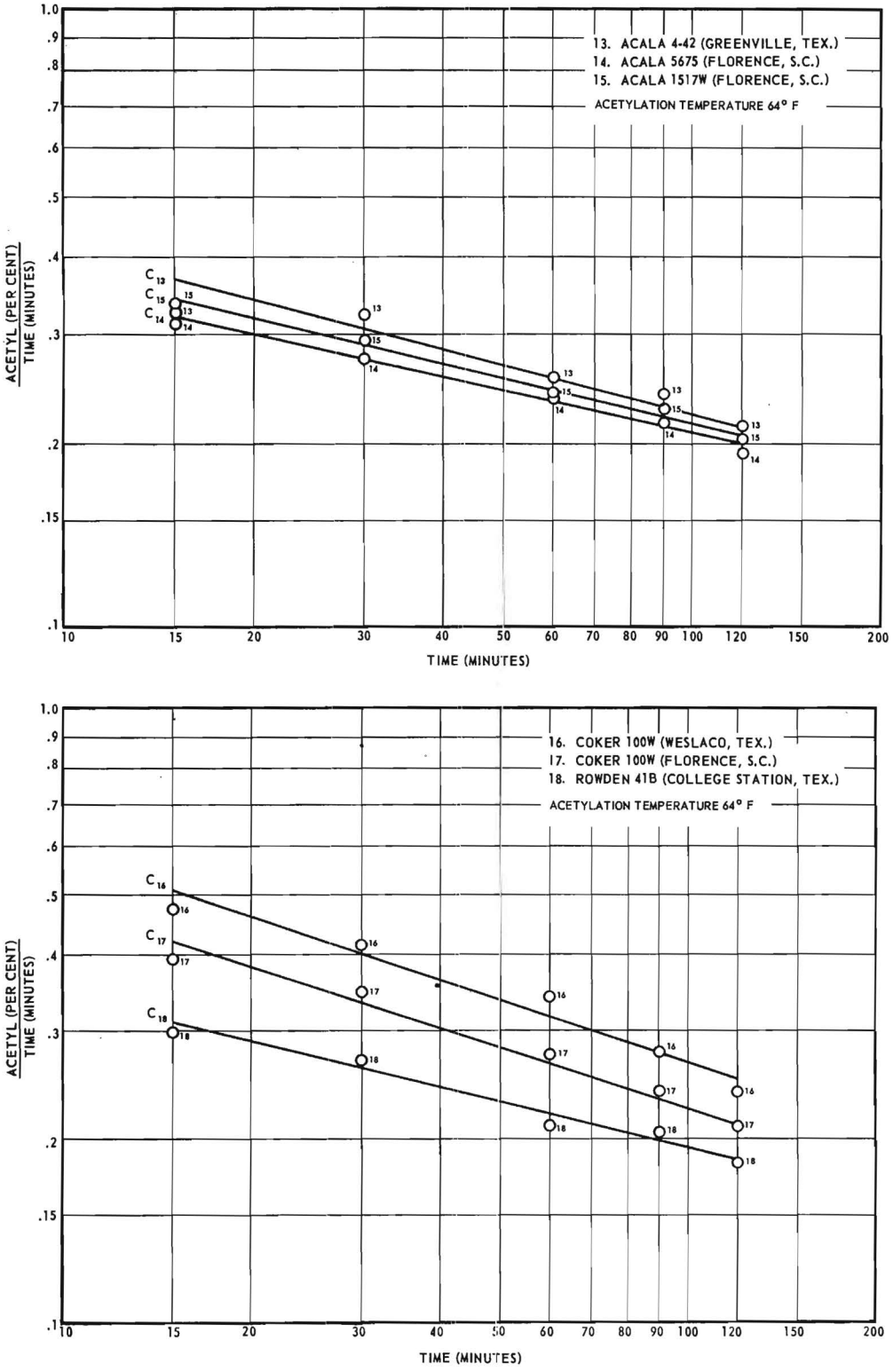

Figure 9. The Change in Rate of Acetylation with Time.

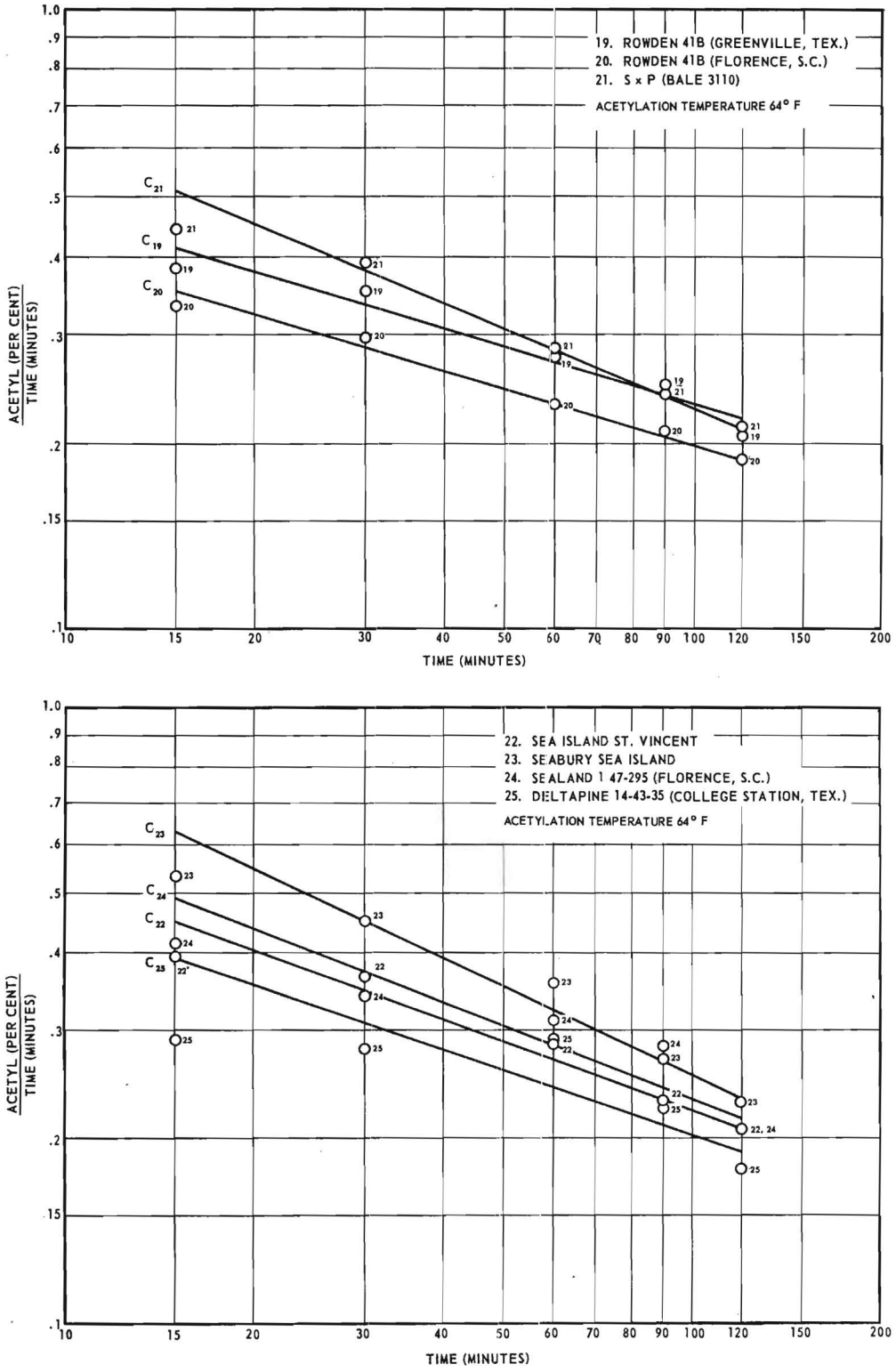

Figure 10. The Change in Rate of Acetylation with Time.

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 7

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

DECEMBER 20, 1953

UNITED STATES DEPARTMENT OF AGRICULTURE AGRICULTURAL RESEARCH ADMINISTRATION BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY	REPORT TO SOUTHERN REGIONAL RESEARCH LABORATORY NEW ORLEANS, LOUISIANA

CONTRACT PROJECT REPORT

CONTRACTING ORGANIZATION	PROJECT LEADERS	
Georgia Tech Research Institute	James L. Taylor	
DEPARTMENT	USDA DESIGNATED REPRESENTATIVE	
Research	Charles F. Goldthwait	
DIVISION	CONTRACT NUMBER	INITIATION DATE
Chemical Sciences	A-1s-33460	March 20, 1952
LOCALITY	REPORT NUMBER	PERIOD COVERED
Atlanta, Georgia	Seven	Sept. 20 - Dec. 20, 1953

TYPE OF REPORT: PROGRESS (X) PHASE ( ) ANNUAL ( ) TERMINATION ( )

PROJECT TITLE:

The Partial Acetylation of Cotton

ABSTRACT OF PROGRESS

Several varieties of scoured and unscoured cotton were extracted with glacial acetic acid, benzene, ethyl alcohol, chloroform, hot water and 0.5% solution of ammonium oxalate. Even mild scouring removed measurable quantities of extractable materials such as pectins and gums. The largest amounts were removed from immature cottons.

Six cotton varieties were scoured with (1) boiling water (60 min.) and (2) a solution of 20% NaOH, 2% synthetic soap and 4% trisodium phosphate (120 min. at 255° F); the cottons were acetylated (30, 60 and 90 min. at 64° F); acetyl values were determined and tabulated. Results show a definite increase in acetylation reactivity for more mature scoured cottons. There is no practical difference in reactivity between cottons scoured with hot water and those scoured with strong alkali.

X-ray diffraction patterns of three varieties of unscoured and scoured cottons show that there is no apparent change in fiber structure corresponding to the large increase in acetylation reactivity of scoured samples.

In order to determine maturity values, 22 cottons were differentially dyed with a mixture of a red dye and a green dye. For qualitative results the dyed samples were classified by color; immature cotton dyed green, mature dyed red. A photovolt reflectometer was used to measure the color of these dyed samples quantitatively. The colorimeter available was not sensitive enough to differentiate between samples relatively close in maturity. Arealometer measurements of 24 cottons also gave maturity values.

The per cent moistures in samples conditioned at 15, 35, 50 and 85% relative humidity (70° F) were redetermined. The new values showed a somewhat (approximately 1%) higher moisture content than the previous measurements.

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

PROGRESS REPORT NO. 7

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR and ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

DECEMBER 20, 1953

TABLE OF CONTENTS

This Report Contains 15 Pages

LIST OF TABLES

I. WORK PROGRAM

During the past quarter the following work was completed.

A. Several scoured and unscoured cottons were extracted using as solvents glacial acetic acid, benzene, ethyl alcohol, chloroform, hot water and a solution of 0.50 per cent ammonium oxalate. These same cottons were also analyzed for per cent α-cellulose content.

B. Additional studies of the effect of scouring on the rate and degree of acetylation were completed. The six cottons selected previously, Memphis, Empire, Bob Shaw, Stoneville 2B, Acala 1517, and Lockett 140, were scoured using only hot water for a very mild scour and a boiling solution containing 20 per cent NaOH, 2 per cent Igepon T and 4 per cent Na_3PO_4 based on weight of the cotton. After scouring, the cottons were acetylated for 30, 60 and 90 minutes at 64° F.

C. X-ray diffraction patterns were made of samples of Memphis, Empire and Lockett 140 cottons before and after scouring. These three cottons were tested because of their wide variations in physical properties and rate of acetylation.

D. Differential dyeings were made of 22 cottons previously used in acetylation experiments in an attempt to determine maturity by this method.

E. Twenty-four of the cottons acetylated previously were tested with an arealometer and the A and D values, which are related to fiber fineness and maturity, were obtained.

F. A recheck was made of the per cent moisture in samples of Memphis, Empire, Bob Shaw, Stoneville 2B, Acala 1517 and Lockett 140 cottons conditioned at 15, 35, 50 and 85 per cent relative humidity at 70° F.

II. EXPERIMENTAL WORK

A. Solvent Extraction of Cottons

Three cottons (Memphis, Empire and Lockett 140) having widely different properties and acetylation rates were selected for the determination of per cent extractables using as solvents chloroform, benzene, 95 per cent ethyl alcohol, distilled water, glacial acetic acid and a 0.50 per cent ammonium oxalate solution.

Except for the ammonium oxalate extraction, duplicate samples of cotton were weighed accurately and placed in paper extraction thimbles; the thimbles were then enclosed in a Soxhlet extraction apparatus. The solvent was added and refluxed for four hours. After the reflux period the solvent, containing the extracted material, was evaporated to dryness. This weighed residue was then reported as per cent solvent-soluble material based on the dry weight of the cotton. The extractions with 0.5 per cent ammonium oxalate solution were made by boiling duplicate samples of accurately weighed cotton for four hours in a reflux apparatus. The cottons were then washed, dried and reweighed, and the per cent of the materials removed by the ammonium oxalate was calculated from the difference in weight from the original sample. Table I lists the various cottons and the per cent extract for the various solvents.

The per cent wax in nine different unscoured cottons was also determined by a method involving extraction of the cotton in a Soxhlet apparatus with 95 per cent ethyl alcohol and then extracting the alcohol with chloroform.[1] The chloroform containing the wax from the cotton was evaporated and the per cent wax calculated. These results are presented in Table II.

---

[1] Conrad, C. M., Ind. and Eng. Chem. 16, 745 (1944).

Determinations of α-cellulose content[2] in Memphis, Empire and Lockett 140 cottons were made by the following method:

Approximately 3 grams of cotton of known moisture content were covered with 35 ml. of NaOH solution containing 17.5 ml. of C. P. NaOH per 100 grams of solution. A total of 40 ml. more of alkali was added in 10-ml. portions over a period of ten minutes with occasional agitation. After 45 minutes, 75 ml. of distilled water were mixed with the mass which was promptly filtered through a Gooch crucible and washed with 750 ml. of distilled water. The mat in the crucible was soaked for 10 minutes in 10 per cent acetic acid to remove adsorbed alkali and then washed free of acid with distilled water. The remaining material, which is α-cellulose, was dried for six hours at 105° C. The weight was determined and the per cent α-cellulose calculated from the original weight of the sample. The amounts of α-cellulose for the various cottons are shown in Table I.

B. Effect of Scouring on Acetylation of Cotton

To present a more complete picture of the effect of scouring cotton on rate and degree of acetylation, six cottons which have been used in previous scouring studies were scoured by the following methods.

1. Hot Water

Seventy-five grams of each cotton were placed in a one-pound-package-dyeing machine; water was added, heated to the boil and circulated at the boil for 60 minutes. The cotton was then removed, extracted, dried and conditioned at 65 per cent relative humidity at 70° F. prior to presoaking for acetylation.

---

[2] Ott, Emil (Editor), High Polymers, Volume 5, Cellulose and Cellulose Derivatives. Interscience Publishers, Inc., New York, 1946, p. 130.

2. Strong Alkali

Seventy-five grams of each cotton were scoured in a 22:1 bath of 20 per cent NaOH, 2 per cent Igepon T and 4 per cent Na_3PO_4 based on weight of the cotton. This scour was carried out under pressure and at approximately 255° F. for 120 minutes. After scouring, the cotton was washed in hot and then in cold water for approximately 30 minutes; then it was removed, extracted, dried and conditioned at 65 per cent relative humidity at 70° F. prior to acetylation.

The scoured and conditioned cottons were presoaked in glacial acetic acid and acetylated at 64° F. for 30, 60 and 90 minutes, respectively, using the procedure described in Progress Report No. 4.

Analyses were made on all of the acetylated cottons to determine per cent moisture and per cent acetyl. The results of these determinations are presented in Table III.

C. X-ray Diffraction

The three cottons, Memphis, Empire and Lockett 140, selected for solvent extraction were also studied with a flat-plate x-ray diffraction camera. A copper anode was used and the x-ray machine was operated at 40 kilovolts and 20 milliamperes. The collimation was provided by two pinholes, the second being 0.025 inch in diameter. A bundle of the cotton fibers about two millimeters in diameter was placed over the second pinhole. The photographic plate was placed approximately five centimeters from the specimen. This experimental arrangement was used for all samples. The exposure time for each sample was approximately two hours, and identical developing procedures were used.

). Differential Dyeings

Approximately three grams each of twenty-two cottons remaining from the original twenty-five used in the study of acetylation reactions were

differentially dyed[3] with Diphenyl Fast Red 5 BL Supra I (Geigy) and Chlorantine Fast Green BLL (Ciba).

The weighed cotton was placed in a boiling 40:1 bath containing 1.2 per cent red dye and 2.8 per cent green dye calculated from the weight of the sample. After 15 minutes at the boil, the cotton was removed and 2.5 per cent of its weight of C. P. NaCl was added. The cotton was re-entered and boiled for another 15 minutes; then it was removed and another 2.5 per cent NaCl was added; the fibers were replaced and boiled for a total of 45 minutes. The sample was then washed in two changes of cold distilled water in proportion of 50 parts to 1 of cotton. The cotton was held in boiling water, for exactly 30 seconds with agitation, removed and pressed to remove the water. In order to compare colors, the dyed samples were ground in a Wiley mill; after this the resulting powder was dispersed in a 40:1 volume of a one per cent sodium carboxy-methyl cellulose solution and filtered through a Gooch crucible. The resulting pad was dried and mounted on white cardboard for comparison.

E. Arealometer Measurements

Through the cooperation of the Fiber Research Laboratory of the University of Tennessee, arealometer measurements were made on 24 of the 25 cottons used in acetylation experiments and reported in Progress Report No. 6. These 24 cottons represented all the samples available.

The arealometer is a null-air-flow instrument designed to measure a fineness characteristic called specific area. Specific area, A, is defined as the external area per unit volume of fiber and is a measure of the fineness of the fiber under normal compression. The increase, D, of the apparent specific

- - - - -

[3] Goldthwait, C. F., Smith, H. O., Barnett, M. P., Textile World 97, 105 (July, 1947).

area, at a much higher compression is a measure of the immaturity of the fiber being tested. The A and D values for the cottons tested are presented in Table IV.

F. Moisture Determinations

Since there was some question as to the accuracy of the moisture determinations made using the Brabender moisture tester and reported previously,[4] it was thought advisable to recheck the moisture by a more exact method. The six cottons used previously to study the effect of moisture conditioning on rate and degree of acetylation were conditioned approximately 100 hours over H_2SO_4 solutions adjusted to give 15, 35, 50 and 85 per cent relative humidity at 70° F. The moistures were determined by carefully and quickly weighing approximately one gram of cotton in a tared weighing bottle. The unstoppered bottle with cotton was then dried approximately three hours at 220° F., the top was replaced and the sample placed in a desiccator. After cooling, the sample and bottle were reweighed and the moisture calculated. The results of the moisture determinations are found in Table V.

III. DISCUSSION OF RESULTS

An analysis of the data from the solvent extraction experiments (Table I) shows that scouring removes a measurable amount of the extractable materials. It should also be noted that the per cent of these solvent extractables is, in general, greater for the less mature cotton samples than for the mature. However, there appears to be little evidence of any drastic difference in the scoured and unscoured cotton which would indicate or explain the greatly increased reactivity of the scoured cottons towards acetylation.

---

[4] Blandin, S. W., A Study of the Effects of Scouring and Moisture Condition on the Rate and Degree of Partial Acetylation of Several Varieties of Cotton Fibers. M. S. Thesis, Georgia Institute of Technology, 1953, p. 45.

Since it was noted that scouring cotton samples prior to acetylation greatly increased the rate and degree of acetylation, it was thought that an x-ray diffraction picture might show whether this increase in reactivity could be related to easily observable changes in the crystal structure of the cottons. Careful examination of the resulting x-ray films for scoured and unscoured samples of the same cotton shows no observable change in structure. Although this eliminates the possibility of any major structural change in the cellulose fiber, it does not eliminate the possibility that some minor change may have occurred which was not revealed by the x-ray diffraction method used.

The additional scouring experiments, in general, show the same results as previous studies, namely, any scouring action greatly increases the reactivity of the mature cottons but has very little effect on the rate of acetylation of the immature cottons tested.

It should also be noted that there is practically no difference in rates of acetylation between the cottons scoured with the strong alkaline solution and those scoured at the boil in water. This is important since a water scour could provide an inexpensive method of obtaining cotton of approximately equal reactivity for partial acetylation on a commercial scale.

The technique of direct dyeing with the combination of two dyes, as described in the experimental work section of this report, differentiates between mature and immature fibers. The terms "mature" and "immature fibers" refer to thick-walled and thin-walled fibers, respectively. The immature fibers dye the green while the mature fiber dye red, using the dyes selected. Therefore, any mixture of mature and immature fibers should have a corresponding color value as a result of a red and green mixture.

The dyed cottons have been arranged by visual inspection in order of color from green to red, but attempts to obtain quantitative differences between dyed cottons were unsuccessful when tested with a photovolt reflectometer.

The arealometer method for determining maturity values for cotton fibers is based on the theory that the immature fibers are collapsed and, therefore, when compressed, tend to twist so that the broad sides are perpendicular to the air flow.[6] This tendency is measured by an increase in the apparent specific area or a D value. The immaturity value was calculated using the following experimental relation:

$$I \text{ (Immaturity)} = \sqrt{0.07\,D + 1},$$

and the per cent maturity was calculated by:

$$M \text{ (Maturity)} = 150.5 - 38.1\,I.$$

Table IV shows the values for A and D which are measured in $mm.^2/mm.^3$ and the calculated "immaturity ratio" I, as well as the per cent maturity values measured by the NaOH and arealometer methods. The maturity values of both methods presented in this table will be used later in correlation studies.

The moisture rechecks for cotton conditioned at various per cent relative humidities posed the problems of the length of time necessary to ensure complete moisture equilibrium and the transfer and initial weighing of the sample without either loss or gain in moisture. The method using weighing bottles was checked against the Brabender method, and the results indicate that the weighing-bottle method is more accurate for analytical work. The results of these moisture determinations are shown in Table V.

- - - - -

[6] Hertel, K. L., and Craven, C. J., Textile Research Journal 21, 765 (1951).

IV. FUTURE PROGRAM

During the next quarter the experimental work will be consolidated. The work will also include the preparation of a final report.

Respectfully submitted:

James L. Taylor
Project Director

Alton R. Colcord, Jr.
Research Assistant

Approved:

Herschel H. Cudd, Director
Engineering Experiment Station

V. APPENDIX

TABLE I

SOLVENT EXTRACTION OF SCOURED* AND UNSCOURED COTTONS

Cotton	α-Cellulose (Per Cent)	Per Cent Extract Using					
		Distilled H_2O	Glacial Acetic	Chloro-form	Benzene	Ethyl Alcohol	Ammonium Oxalate Solution
Unscoured:							
Memphis	95.3	2.65	6.10	0.93	1.00	2.20	4.50
Empire Bale 92	97.05	2.40	5.30	0.51	0.63	1.40	3.50
Lockett 140	97.35	2.20	5.10	0.37	0.50	1.20	3.80
Scoured:							
Memphis	98.55	1.50	2.75	0.80	0.84	1.05	0.74
Empire Bale 92	99.00	1.20	2.05	0.38	0.43	0.71	0.48
Lockett 140	99.25	1.10	1.85	0.31	0.35	0.52	0.41

*Scoured with a boiling solution of 1 per cent Duponal RA and 1.5 per cent tetrasodium pyrophosphate (percentages based on weight of cotton).

TABLE II

WAX CONTENT DETERMINED BY ALCOHOL-CHLOROFORM EXTRACTION METHOD

Cotton	Per Cent Wax	Cotton	Per Cent Wax
Memphis	1.40	Coker 100W (Westlaco, Texas)	0.64
Empire Bale 92	0.77	Coker 100W (Florence, S. C.)	0.56
Lockett 140	0.56	Sea Island St. Vincent	1.00
Acala 4-42 (Greenville, Texas)	0.61	Sealand 1	0.67
Acala 5675 (Florence, S. C.)	0.57		

TABLE III

ACETYL AND MOISTURE CONTENTS* OF SCOURED COTTONS
ACETYLATED AT 64° F. FOR VARIOUS LENGTHS OF TIME

Cotton	30 Minutes		60 Minutes		90 Minutes	
	Per Cent H_2O	Per Cent Acetyl	Per Cent H_2O	Per Cent Acetyl	Per Cent H_2O	Per Cent Acetyl
Hot Water Scour:						
Memphis	4.1	16.2	3.5	21.8	3.6	23.9
Empire Bale 92	4.1	14.2	3.4	19.4	3.4	22.7
Bob Shaw	4.2	14.1	3.4	19.0	3.3	21.8
Stoneville 2B Bale 249290	4.1	14.4	3.4	20.0	3.5	22.2
Acala 1517	4.1	15.0	3.4	20.6	3.4	22.9
Lockett 140	4.2	13.6	3.6	19.0	3.4	21.8
Strong Alkali Scour:						
Memphis	3.7	16.4	3.1	19.7	3.3	23.7
Empire Bale 92	3.9	15.1	3.2	20.0	3.1	21.5
Bob Shaw	3.8	13.9	3.2	19.0	3.2	20.9
Stoneville 2B Bale 249290	3.8	14.9	3.2	19.6	3.1	22.1
Acala 1517	3.4	15.8	3.3	20.6	3.1	22.4
Lockett 140	4.0	14.7	3.3	19.5	3.3	22.6

*Moisture content was determined after conditioning the samples at 70° F. and 65 per cent relative humidity.

TABLE IV

AREALOMETER MEASUREMENTS AND MATURITY VALUES
FOR TWENTY-FOUR COTTONS USED FOR PARTIAL ACETYLATION EXPERIMENTS

Cotton	A ($mm.^2/mm.^3$)	D ($mm.^2/mm.^3$)	I	Maturity[1] (Per Cent)	Maturity[2] (Per Cent)
Empire Bale 92	502	42	1.985	72	75
Sea Island	587	40	1.949	86	76
Indian J & J	418	31	1.780	93	83
S x P Bale 3109	514	19	1.526	86	92
Hopi Acala 50	415	10	1.304	95	101
Pima 32	559	31	1.780	84	83
Memphis	---	--	---	38	--
Lockett 140	510	41	1.967	92	76
Coker 100W (Florence, S. C.)	422	24	1.637	86	88
Acala 5675 (Florence, S. C.)	444	28	1.720	82	85
Deltapine	513	38	1.913	72	78
Acala 1517	468	27	1.700	86	86
Stoneville 2B Bale 249290	505	38	1.913	80	78
Bob Shaw	396	20	1.549	--	92
Stoneville 2B Bale 616654	483	35	1.857	79	80
Sealand 1	498	30	1.761	82	83
Rowden 41B (Florence, S. C.)	357	22	1.594	85	90
Coker 100W (Westlaco, Texas)	457	27	1.700	77	86
Rowden 41B (College Station, Texas)	438	29	1.740	81	84
Acala 4-42 (Greenville, Texas)	482	28	1.720	77	85
S x P Bale 3110	489	20	1.549	86	92

(Continued)

TABLE IV (Continued)

AREALOMETER MEASUREMENTS AND MATURITY VALUES
FOR TWENTY-FOUR COTTONS USED FOR PARTIAL ACETYLATION EXPERIMENTS

Cotton	A ($mm.^2/mm.^3$)	D ($mm.^2/mm.^3$)	I	Maturity[1] (Per Cent)	Maturity[2] (Per Cent)
Sea Island St. Vincent	636	53	2.170	80	68
Rowden 41B (Greenville, Texas)	396	22	1.549	87	90
Acala 1517W (Florence, S. C.)	462	21	1.572	85	91

[1] Maturity values determined by NaOH method.

[2] Maturity values determined by arealometer.

TABLE V

COMPARISON OF MOISTURE CONTENTS OF RAW COTTON
CONDITIONED AT VARIOUS PER CENT RELATIVE HUMIDITIES

Cotton	Per Cent Moisture at 15 Per Cent Relative Humidity		35 Per Cent Relative Humidity		50 Per Cent Relative Humidity		85 Per Cent Relative Humidity	
	Old	New	Old	New	Old	New	Old	New
Memphis	4.1	4.71	4.2	5.69	5.0	5.89	5.8	10.06
Empire Bale 92	3.7	4.57	4.2	5.71	5.1	6.08	5.8	10.13
Bob Shaw	3.5	4.19	4.1	5.32	5.0	6.35	5.4	9.80
Stoneville 2B Bale 249290	4.2	4.54	4.3	5.48	5.3	6.07	5.9	9.09
Acala 1517	4.1	3.42	4.3	5.14	5.3	6.31	6.0	9.45
Lockett 140	3.4	3.52	3.9	5.10	5.2	6.61	5.9	9.98

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

FINAL REPORT

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR AND ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

MARCH 20, 1954

UNITED STATES DEPARTMENT OF AGRICULTURE AGRICULTURAL RESEARCH ADMINISTRATION BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY	REPORT TO SOUTHERN REGIONAL RESEARCH LABORATORY NEW ORLEANS, LOUISIANA

CONTRACT PROJECT REPORT

CONTRACTING ORGANIZATION	PROJECT LEADERS	
Georgia Tech Research Institute	James L. Taylor	
DEPARTMENT	USDA DESIGNATED REPRESENTATIVE	
Research	Charles F. Goldthwait	
DIVISION	CONTRACT NUMBER	INITIATION DATE
Chemical Sciences	A-1s-33460	March 20, 1952
LOCALITY	REPORT NUMBER	PERIOD COVERED
Atlanta, Georgia	Final	March 20, 1954

TYPE OF REPORT: PROGRESS ( ) PHASE ( ) ANNUAL ( ) TERMINATION (X)

PROJECT TITLE:

The Partial Acetylation of Cotton

ABSTRACT OF PROGRESS

The purpose of this investigation was to determine the relationships between cottons of different chemical and physical properties and their response to acetylation.

Twenty-five different cottons in their natural state, representing a wide range of properties, were selected from samples furnished by the Southern Regional Research Laboratory in New Orleans, La. All of the 25 cottons were acetylated at a constant temperature of 64° F for different periods of time varying from 15 to 120 minutes duration. The acetylations were made in a one-pound stainless-steel package dyeing machine which was modified to maintain close temperature control of the acetylating mixture consisting of three parts glacial acetic acid, one part acetic anhydride and a small amount of perchloric acid catalyst.

Studies were also made to determine the effects of scouring treatments on various cottons prior to acetylation. The results of these studies showed that scouring increased the rate and degree of acetylation and minimized the inherent differences in cottons toward acetylation.

Experiments to determine the effects of different moisture contents of raw cottons on the rate and degree of acetylation showed that cottons conditioned at the higher humidities displayed an increase in the rate and degree of acetylation.

Mild scouring was found to remove about half the extractable materials from cotton; based upon extractions with six different solvents on three cottons (Memphis, Empire and Lockett 140) having widely different properties. Details were given

in Quarterly Report No. 7.

The wax content of nine cottons was found to vary from 0.56 per cent for Lockett 140 to 1.40 per cent for Memphis, as determined by means of the alcohol-chloroform method.

Detailed data from these experiments are presented in tabular form in this report. Statistical analyses of the acetylation data and correlation data are also discussed and results presented in tables. The correlation studies show that the degree and rate of acetylation cannot be predicted with accuracy from the physical and chemical properties of the cottons employed in this study.

However, several significant correlations were established among varieties and acetylation conditions and degree of acetylation. The individual correlations are listed under general conclusions and detailed in Table VI.

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

FINAL REPORT

PROJECT NO. 208-156

PARTIAL ACETYLATION OF COTTON

By

JAMES L. TAYLOR AND ALTON R. COLCORD, JR.

- o - o - o - o -

CONTRACT NO. A-1s-33460

THE UNITED STATES DEPARTMENT OF AGRICULTURE
SOUTHERN REGIONAL RESEARCH LABORATORY

- o - o - o - o -

MARCH 20, 1954

TABLE OF CONTENTS

(Continued)

TABLE OF CONTENTS (Continued)

This Report Contains 35 Pages

LIST OF TABLES

I. INTRODUCTION

Previous work at the Southern Regional Research Laboratory[1] has revealed that cotton fibers can be chemically modified to improve their resistance to microbiological rotting, mildewing and heat degradation. Partially acetylated cotton is an example of such a modification. However, various cottons differ in their response to acetylation, i.e., they have different rates of acetylation. At present, there are no known tests, other than determination of acetyl content, for determining differences in the extent of acetylation for various cottons. Qualitatively, it has been found that the degree of maturity of cotton fibers has an effect on the acetylation characteristics; however, this is undoubtedly not the entire answer. Other characteristics of cotton in its natural state, as well as in its preparation for acetylation, may influence the reaction rate. Some of these properties are listed here:

1. Cotton variety
2. Area of growth
3. Fiber maturity
4. Fiber fineness
5. Fiber strength
6. Per cent crystalline cellulose
7. X-ray angle (crystallite orientation)
8. Alcohol-soluble content
9. Wax content
10. Ash constituents
11. Moisture content

---

[1] Cooper, Albert S., Voorhies, Samuel T., Jr., Buras, Edmund M., Jr., and Goldthwait, Charles F., "Partial Acetylation of Cotton," Textile Industries 116, No. 1, 97 (1952).

Since very little investigation had been conducted on the relationship of the previously mentioned variables and the rate and degree of acetylation of cotton fibers, a research program was set up at the Engineering Experiment Station at Georgia Institute of Technology under the sponsorship of the U.S.D.A. through the S.R.R.L. in New Orleans.

By mutual agreement, 25 cottons which represent wide differences in variety, area of growth, chemical and physical properties were selected. All of these cottons were acetylated at a constant temperature for varying lengths of time, ranging from 15 to 120 minutes. Additional acetylation studies were made to determine the effects of moisture-content, alkali-soluble, water-soluble, alcohol-soluble and acetic-acid-soluble constituents. Studies were also made to ascertain the effects of temperature and time of presoaking of the various cottons prior to acetylation. Finally, statistical analyses of the acetylation data were made.

These data will be presented in the section on experimental results and statistical analyses.

II. EXPERIMENTAL WORK

A. Materials

As mentioned above, 25 different cottons were selected for experimentation. This was an arbitrary number and had no particular significance to the problem other than an attempt to study as many types of cottons as possible in the time alloted for the investigation. The cottons represented differences in maturity, fiber fineness, variety, crystallite orientation, strength and area of growth. Each cotton was mechanically cleaned and blended by a Shirley Analyzer prior to acetylation treatments, thus eliminating variations due to

nonuniformity of sample. A list of the cottons used and their properties are given in Table I.

B. Acetylating Equipment

A Morton one-pound-package-dyeing machine was modified to handle raw-stock fibers. After trial acetylations, it was concluded that cooling by means of circulating ice water around the acetylating chamber of the machine was insufficient to maintain a constant acetylation temperature throughout the acetylating solution. Thus it became necessary to change the circulating system to permit the acetylating mixture to be cooled by passing it through a series of stainless-steel coils mounted in a tank filled with ice water. Details of this equipment may be seen in Figures 1 and 2.

C. Methods of Analyses

1. Acetyl Content

Duplicate 1.0-to 1.5-gram samples were selected from the samples of acetylated cotton which had been conditioned at 65 per cent relative humidity and 70° F, and these samples were ground in a Wiley Mill. The samples were then transferred into tared 250-milliliter Erlenmeyer flasks, and the gross weights accurately determined on an analytical balance. The exact net weights of the ground samples were then calculated and converted into dry weights using the per cent moisture figures obtained for each sample. Fifty milliliters of 75 per cent methyl alcohol were then added to each flask, and the flasks with stoppers loosely inserted were heated for 15 to 20 minutes at 140 F. To each flask fifty milliliters of an approximately 0.5 normal sodium hydroxide solution were then added, after which the flasks were heated again at 140 F for one hour. The flasks were then tightly stoppered, and the samples were allowed to saponify overnight. The rubber stoppers and the walls of each flask were then

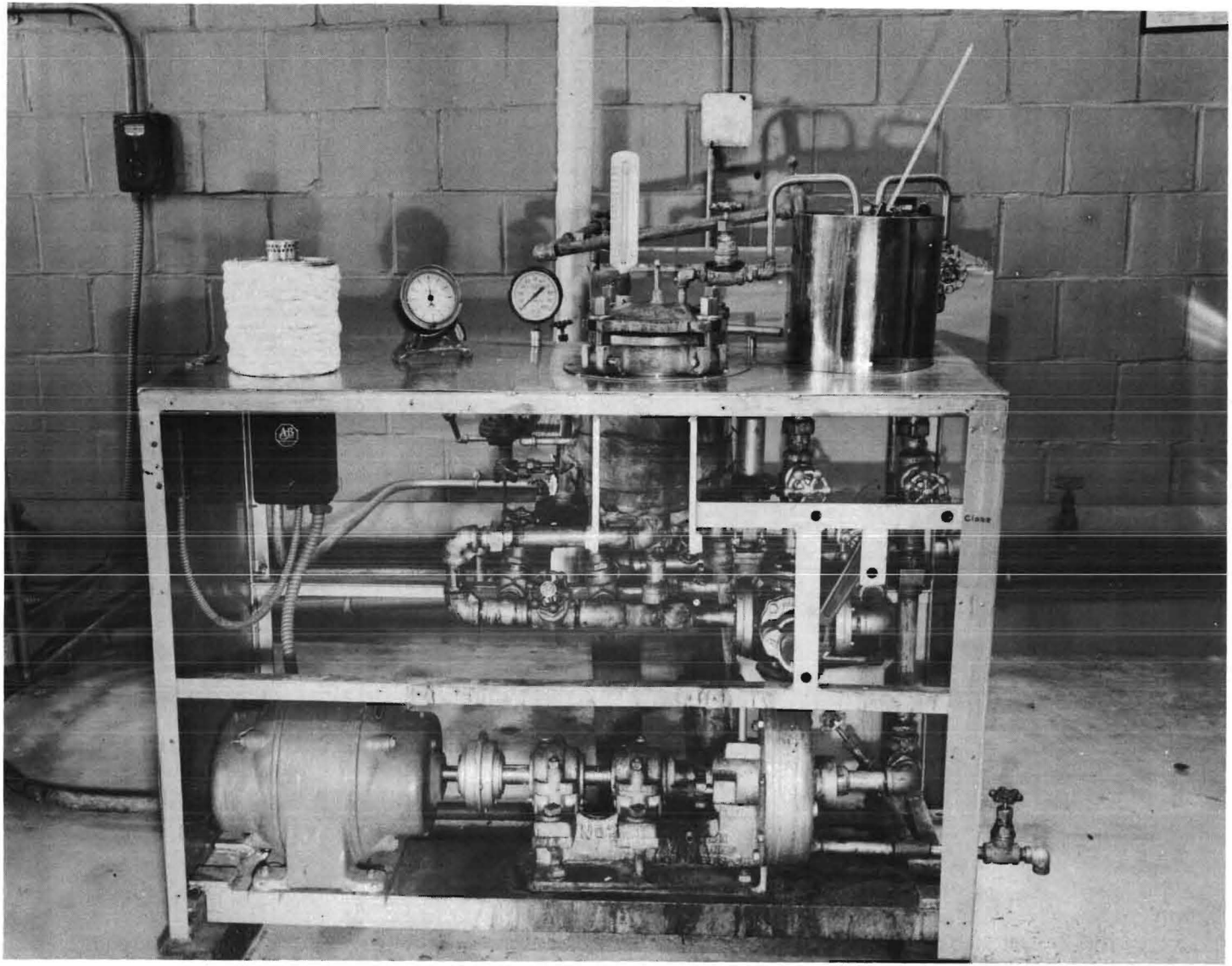

Figure 1. Front View of the Acetylation Machine.

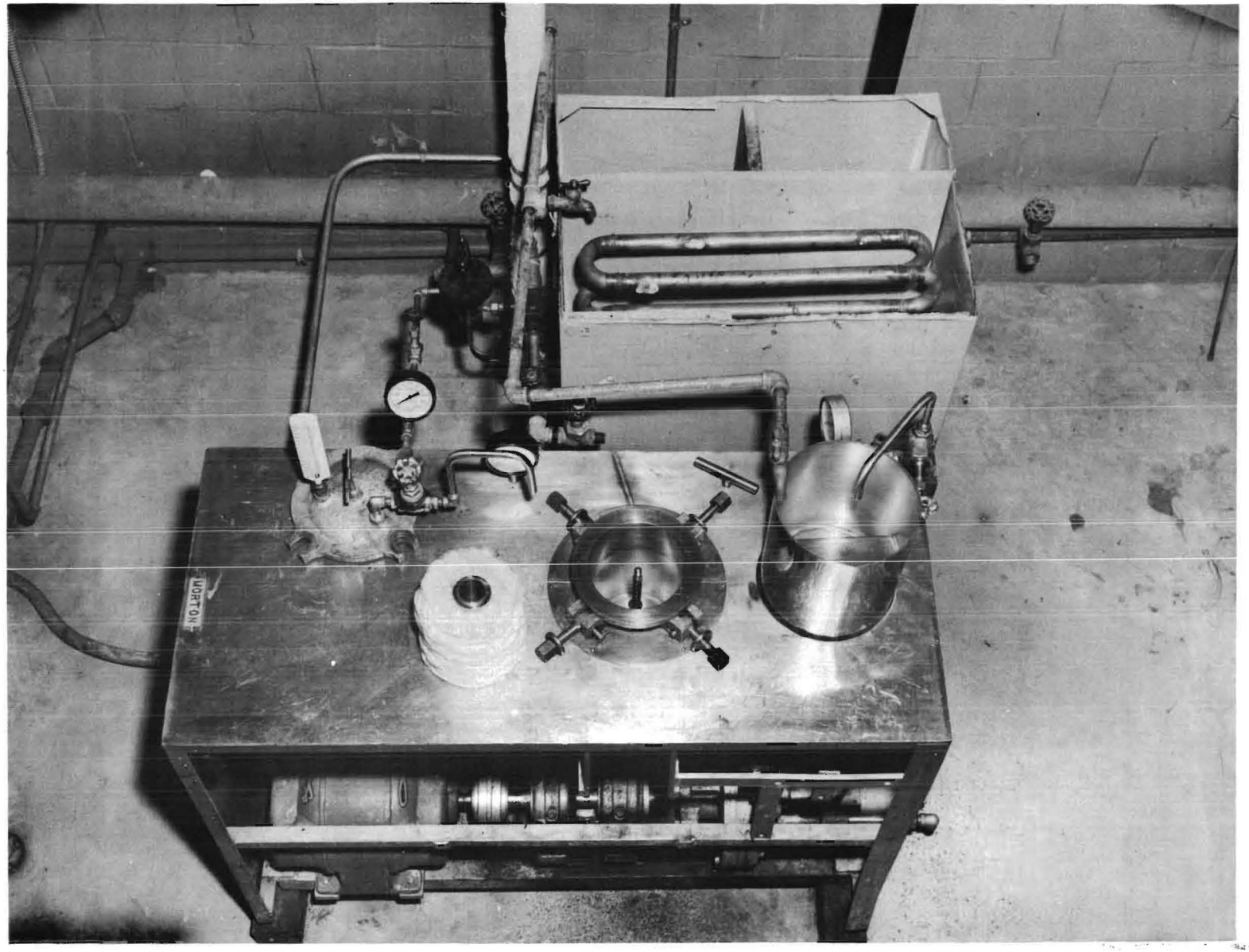

Figure 2. Top View of the Acetylation Machine.

washed down with distilled water, and two or three drops of phenolphthalein were added for use as an indicator in titration. Standardized approximately 0.5 normal hydrochloric acid solution was used for the titrations. Two blanks of unacetylated cotton were included with each set of samples and were treated in the same manner as the other samples. Two determinations for acetyl content were made for each sample of acetylated cotton, and the average value was accepted if the two determinations were within 0.5 per cent of each other. The acetyl content was calculated using the following formula:

$$\text{Per cent acetyl} = \frac{(a-b) \times N \times 0.04302 \times 100}{d}$$

where

a = Volume of HCl in milliliters required to titrate the blank,

b = Volume of HCl required to titrate the sample,

N = Normality of HCl solution,

d = Dry weight of the sample in grams, and

0.04302 = Milliequivalent weight of the acetyl group.

2. Moisture Content

The per cent moisture of each variety of cotton before and after acetylation was determined at standard conditions.

3. Fiber Strength

Selected samples of raw cotton and acetylated cottons were tested at standard conditions for strength index according to the method described in A.S.T.M. Standards on textile materials D 414-49T. The Pressley cotton fiber-strength tester was used in making these determinations.

4. Fiber Fineness

The fiber fineness of each sample of raw cotton was determined at

standard conditions prior to treatment by means of the Sheffield Micronaire, and in accordance with the procedure recommended by the manufacturer of the instrument.

Fiber fineness was also determined from arealometer data furnished by the Fiber Research Laboratory of the University of Tennessee.

5. Maturity

The per cent maturity values of each variety of raw cotton was determined by swelling the samples in 18 per cent sodium hydroxide solution according to the method described in A.S.T.M. D-414-49T. These values were furnished by the Southern Regional Research Laboratory. Per cent maturity was also calculated from the arealometer data furnished by Fiber Research Laboratory, University of Tennessee.

6. Crystallite Orientation

The crystallite orientation data as measured by x-ray angle of each variety of raw cotton was also furnished by the Southern Regional Research Laboratory.

7. Evenness of Acetylation

Two- to three-gram samples of each acetylated cotton were dyed with a solution containing 4 per cent Chloratine Fast Blue 3 RLL, 4 per cent Celliton Fast Yellow RRA and 5 per cent Triton X-100, a wetting agent, calculated on the weight of the samples. The liquor ratio of the dye bath was 70:1. After dyeing for 30 minutes, 50 per cent anhydrous sodium sulfate based on the total sample weight was added, and dyeing was continued for another 30 minutes. The dyed samples were then washed well with cold water and dried in the hot-air-circulating oven. Since the yellow dye is an acetate dye which will not stain cotton, and the blue dye is a direct cotton dye which will not

stain cellulose acetate, the well acetylated portions of the fiber dye to a full shade of yellow and the unacetylated portions, if any, dye blue. Portions acetylated to an intermediate degree may appear to be green. The depth of shade indicates visually the degree of acetylation. However, the primary function of the dye test in this investigation was to indicate the evenness of acetylation.

8. Solvent Extraction

Several selected cottons were extracted using as solvents, chloroform, benzene, 95 per cent ethyl alcohol, distilled water, glacial acetic acid and 0.50 per cent ammonium oxalate solution.

Except for the ammonium oxalate extraction, duplicate samples of cotton were weighed accurately and placed in paper extration thimbles; the thimbles were then enclosed in a Soxhlet extraction apparatus. The solvent was added and refluxed for four hours. After the refluxing period, the solvent containing the extracted material was evaporated to dryness. This weighed residue was then reported as per cent solvent-soluble material based on the dry weight of the cotton. The extractions with 0.5 per cent ammonium oxalate solution were made by boiling duplicate samples of accurately weighed cotton for four hours in a reflux apparatus. The cottons were then washed, dried and reweighed, and the per cent of the materials removed by the ammonium oxalate was calculated from the difference in weight from the original sample.

The per cent wax in nine different unscoured cottons was also determined by a method involving extraction of the cotton in a Soxhlet apparatus with 95 per cent ethyl alcohol and then extracting the alcohol with chloroform.[2]

[2] Conrad, C. M., Ind. and Eng. Chem. 16, 745 (1944).

The chloroform containing the wax from the cotton was evaporated and the per cent wax calculated.

9. Other Special Analyses

Three cottons differing widely in maturity, fineness and wax content were selected for α-cellulose content, ash analyses and wax content as well as x-ray diffractions.

a. α-Cellulose. Determinations of α-cellulose content in Memphis, Empire and Lockett 140 cottons were made by the following method:

Approximately 3 grams of cotton of known moisture content were covered with 35 ml. of NaOH solution containing 17.5 grams of C. P. NaOH per 100 grams of solution. Over a period of ten minutes a total of 40 additional ml. of alkali in 10-ml. portions was added with occasional agitation. After 45 minutes, 75 ml. of distilled water were mixed with the mass which was filtered through a Gooch crucible and washed with 750 ml. of distilled water. The mat in the crucible was soaked for 10 minutes in 10 per cent acetic acid to remove adsorbed alkali and then washed free of acid with distilled water. The remaining material, which was α-cellulose, was dried for six hours at 105° C. The weight was determined and the per cent α-cellulose calculated from the original weight of the sample.

b. Ash Analysis. For the determination of ash content an approximately 10-gram sample of each cotton was accurately weighed in a platinum crucible. The crucible and cotton were then placed in a muffle furnace and ashed at approximately 650° C. After cooling in a desiccator, the sample was weighed and the per cent ash calculated.

The ash from duplicate samples was then combined, and the constituents were determined by spectrographic analysis.

c. X-ray Diffraction. The three cottons, Memphis, Empire and Lockett 140, were studied with a flat-plate x-ray diffraction camera. A copper anode was used and the x-ray machine was operated at 40 kilovolts and 20 milliamperes. The collimation was provided by two pinholes, the second being 0.025 inch in diameter. A bundle of the cotton fibers about two millimeters in diameter was placed over the second pinhole. The photographic plate was placed approximately five centimeters from the specimen. This experimental arrangement was used for all samples. The exposure time for each sample was approximately two hours, and identical developing procedures were used.

D. Acetylation Studies

In order to determine the rate and degree of acetylation of raw cotton for comparison with the physical and chemical data obtained as mentioned previously, the 25 selected cottons were acetylated for 15, 30, 60, 90 and 120 minutes respectively at 64° F.

To demonstrate the effect of scouring on the rate and degree of acetylation, 6 of the 25 cottons representing a wide range of properties were scoured by six different methods. These methods are:

Method I

Cottons treated with boiling water

for 60 minutes.

Dried at 105° C.

Method II

Cottons treated with a solution containing

1.0 per cent Duponol RA

1.5 per cent tetrasodium pyrophosphate

for 30 minutes at the boil.

Rinsed in warm water and dried at 105° C.

Method III

Cottons treated with a solution containing

2.0 per cent sodium hydroxide

1.0 per cent Merpol C

0.5 per cent Duponol RA

0.5 per cent tetrasodium pyrophosphate

for 60 minutes at the boil.

Rinsed in warm water and dried at 105° C.

Method IV

Cottons treated with a solution containing

3.0 per cent sodium hydroxide

0.1 per cent Triton X-100

0.3 per cent tetrasodium pyrophosphate

for 60 minutes at the boil.

Rinsed in warm water and dried at 105° C.

Method V

Cottons treated with a solution containing

5.0 per cent sodium hydroxide

2.0 per cent soap (neutral oleate)

for 90 minutes at the boil.

Rinsed and dried at 105° C.

Method VI

Cottons treated in a solution containing

20 per cent NaOH

2.0 per cent Igepon T

4.0 per cent Trisodium Phosphate

for 120 minutes at 225° F.

Rinsed in warm water and dried at 105° C.

All percentages are based on the weight of the cotton.

Each of the scoured cottons were acetylated for 30, 60, 90 minute periods at 64° F. Data for the acetylation of scoured and unscoured cottons are shown in Table II.

The same six cottons selected for scouring studies were conditioned at 15, 35, 50, 65 and 85 per cent relative humidity and then acetylated for 30, 60 and 90 minutes. These acetylations were performed to show the effect of moisture content of the fiber on the rate and degree of acetylation. In order to produce the desired relative humidities, standard ten-inch desiccators containing various sulfuric acid-water solutions were used as conditioning containers. Six 20-gram samples of cotton were conditioned at one time in each desiccator, each sample representing one of the varieties of cotton chosen

for the study. In order to facilitate rapid transfer of the samples from the humidifying chamber to the presoaking container, thus avoiding an appreciable change in the moisture content of the conditioned cottons during transfer, the samples were arranged in horizontal layers around a vertical wooden spindle with circular stainless-steel screens used to separate the layers. The cotton samples were then placed in the humidifying chamber and allowed to condition for a minimum of 24 hours to assure that moisture equilibrium was reached.

To determine the effect of the presoaking time and presoaking temperature on the degree of acetylation, six varieties of cotton namely, Memphis, Empire, Bob Shaw, Stoneville 2B (Bale 249290), Acala 1517 and Lockett 140, were presoaked for 10, 30, 60, 120 and 240 minutes at 70°, 100°, 130° and 170° F and then acetylated for 45 minutes at 64° F.

Additional studies were made on the same six cottons to show the effect of acetylation temperature on degree of acetylation. The temperatures used were 58°, 64°, 70°, 76° and 82° F respectively. All acetylations were carried out for 45 minutes after presoaking the samples at room temperature for 18 hours.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. The Effects of Chemical and Physical Properties on the Acetylation of Raw Cotton

Table I shows a wide variation in acetyl content for the raw cottons studied. There was little correlation between the acetyl values and the physical and chemical properties of the fibers. This is not true for six selected cottons. For example, in comparing the rates or degrees of

acetylation of Memphis, Empire, Bob Shaw, Stoneville 2B (Bale 249290), Acala 1517 and Lockett 140 with their values of maturity and fiber fineness, there seems to be an obvious relation between per cent maturity and degree of acetylation. Also from comparisons of the per cent wax and acetyl content of the nine cottons for which wax values were determined, it appears that cottons with a high wax content acetylate more readily and to a greater extent than cottons with less wax.

The fact that the comparison of the acetyl content with the physical and chemical properties for the 25 cottons showed no observable relationship indicates that the six cottons selected are probably not representative of the entire group. From the statistical data obtained from the 25 cottons it may be concluded that there is no obvious relationship between rate or degree of acetylation and physical properties measured. However, there seems to be some relation between content wax and the rate or degrees of acetylation of raw cotton, which are also somewhat related to the maturity of the cotton fibers.

B. The Effect of Scouring on the Rate and Degree of Acetylation

The results of the acetylations of six cottons scoured by six progressively severe scours are presented in Table II. It is noted that after scouring, the degree of acetylation for the cottons of high reactivity increases only slightly while the acetyl content of low reactivity cottons increases to a much greater extent. The net result is that any scour tends to minimize the difference in reactivity among these cottons.

The rate of acetylation for the cottons increases slightly from Scour I through Scour V as the severity of the scour is increased. An exception is

noted in the results of Scour VI which is the most severe but gives the least increase in reactivity of all the scours. It may be that scouring under pressure is responsible for the lack of increase in reactivity with increase in severity. However, additional tests should be made before drawing conclusions.

C. The Effect of Moisture Content on Rate and Degree of Acetylation

Acetylating data in Table III give the results of conditioning the cotton under various per cent relative humidities. In general it appears that the increase in the moisture content of the cotton which occurs at increased relative humidity causes an increase in the rate and degree of acetylation.

Cottons conditioned at the low relative humiditives (15, 35) show little difference in acetylation rate. This may be caused by little difference in moisture content or that the moisture content is below a level required to show appreciable effect of the subsequent acetylation reactions.

D. The Effect of Time and Temperature of Presoaking on the Degree of Acetylation

A study of the data presented in Table IV shows that the presoaking temperature has a marked effect on the degree of acetylation.

The duration of presoaking time also shows some effect on degree of acetylation but much less than that of temperature. Thus it can be seen that as the temperature of presoaking was increased, particularly at the shorter presoaking periods, the degree of acetylation rapidly increased. This was especially true in the case of mature cottons such as Acala 1517.

However, it should be noted that although increasing the temperature of presoaking in glacial acetic acid increased the rate of acetylation, it did not affect the maximum degree of acetylation obtained under constant acetylating conditions. The time and temperature of presoaking does not eliminate

the differences between varieties of cotton but does activate the cottons.

E. The Effect of Acetylation Temperature on the Degree of Acetylation.

Early experiments reported in Progress Report No. 3 showed that a variation in acetylating temperatures caused a wide variation in the per cent acetyl content of the acetylated fibers. Therefore, a study was made to determine the effect of temperature on the degree of acetylation. These results are shown in Table V.

From these data it is noted that an increase of 24° F (58° to 82° F) in acetylation temperature approximately doubled the acetyl content of these cottons. It is also evident that the fiber strength is less as the reaction temperature is increased. This loss of strength is probably not excessive at 70° F but an increase to 76° F produces pronounced weakening and a further increase to 82° F so weakened the fiber that reliable strength measurements could not be made.

IV. STATISTICAL ANALYSIS OF ACETYLATION DATA

A. Correlation Studies

In order to fulfill the requirements of part II and part III of the subject contract, a statistical analysis was made of the acetylation data to find some means of predicting the rate and degree of acetylation of raw cotton from its physical or chemical properties.

The first attack on the problem was to obtain a numerical characterization which would represent the acetylation of raw cottons. By plotting the acelytation data several different ways it was found that a plot of the log of the per cent acetyl per minute vs. the log of the acetylation time in minutes resulted in a straight line. Therefore, the acetylation data

TABLE I

PHYSICAL AND CHEMICAL PROPERTIES OF COTTONS USED IN ACETYLATION STUDIES

	Cotton	Area of Growth	Wax Content (%)	Moisture (%)	X-Ray Angle (40%)	Pressley Index	Fiber Finess		Maturity		Per Cent Acetyl					Acetylation* Index
							Aeralometer (gm./in.)	Micronaire (gm./in.)	Aeralometer Method (%)	Alkali Method (%)	15 (min.)	30 (min.)	60 (min.)	90 (min.)	120 (min.)	
1.	Indium J & J			7.2	35.0	7.40	4.93	4.9	83	93	8.57	10.6	17.0	21.8	23.8	-0.475
2.	Sea Island			7.0	36.4	8.66	2.75	2.4	76	86	5.30	8.02	15.5	21.3	23.0	-0.247
3.	Memphis		1.40	7.6	37.9	7.15	--	2.5	--	38	10.1	14.1	20.2	23.9	25.5	-0.533
4.	Empire (Bale 92)		0.77	7.1	34.1	7.30	3.81	3.7	75	72	5.28	8.48	15.4	18.5	22.6	-0.292
5.	Stoneville 2B (Bale 61654)			7.0	--	7.40	3.86	3.9	80	79	4.30	7.33	12.9	17.2	20.7	-0.235
6.	Bob Shaw			7.0	31.3	8.17	4.79	5.1	92	--	3.79	6.84	13.1	16.4	20.4	-0.185
7.	Stoneville 2B (Bale 249290)			7.3	31.2	8.07	3.63	3.2	78	80	7.05	10.5	17.0	20.6	23.7	-0.404
8.	Acala 1517			7.2	29.8	8.93	3.76	4.0	86	86	7.51	11.5	18.0	22.0	25.4	-0.408
9.	S X P (Bale 3109)			7.0	--	8.30	2.81	3.6	92	86	3.21	6.49	11.8	15.7	19.8	-0.131
10.	Lockett 140		0.56	7.2	36.2	7.51	3.68	5.6	76	92	3.91	6.65	12.0	16.5	20.9	-0.190
11.	Pima 32			7.4	29.3	10.50	2.76	2.8	83	84	5.12	9.98	16.5	21.0	25.0	-0.243
12.	Hopi Acala 50			7.5	33.8	8.60	3.68	4.6	101	95	6.32	9.95	16.9	21.1	25.3	-0.325
13.	Acala 4-42	Greenville, Tex.	0.61	7.1	28.8	9.24	4.99	3.9	85	77	4.66	8.30	14.2	19.6	23.2	-0.222
14.	Acala 5675	Florence, S. C.	0.57	7.1	32.6	8.22	4.23	4.2	85	82	5.86	10.4	16.4	21.4	25.1	-0.304
15.	Acala 1517W	Florence, S. C.		7.0	28.0	8.21	3.58	4.0	91	85	5.78	10.6	16.7	22.6	24.8	-0.292
16.	Coker 100W	Weslaco, Tex.	0.64	7.2	33.6	6.96	3.94	4.5	86	77	5.08	8.83	14.5	20.6	24.4	-0.241
17.	Coker 100W	Florence, S. C.	0.56	7.0	35.5	5.91	4.45	4.6	88	86	4.97	8.88	13.9	19.1	22.8	-0.272
18.	Rowden 41B	College Sta. Tex.		6.9	29.6	7.75	4.40	4.8	84	81	4.47	8.04	12.5	18.3	21.7	-0.242
19.	Rowden 41B	Greenville, Tex.		7.1	31.4	7.85	4.79	5.5	90	87	6.67	11.8	17.3	21.9	25.9	-0.361
20.	Rowden 41B	Florence, S. C.		7.0	35.9	6.84	6.09	5.8	90	85	5.95	10.9	16.9	20.7	24.8	-0.326
21.	S X P (Bale 3110)			--	--	8.70	3.14	3.8	92	93	4.43	8.38	17.3	20.2	21.4	-0.207
22.	Sea Island St. Vincent		1.00	7.1	--	10.10	2.60	1.8	68	80	8.03	12.5	21.4	24.2	27.5	-0.384
23.	Seabury Sea Island			--	33.0	--	--	3.1	--	75	6.20	10.2	18.6	20.8	24.8	-0.156
24.	Sealand I	Florence, S. C.	0.67	7.4	31.6	7.71	3.45	3.4	83	82	7.11	12.4	20.4	24.8	28.4	-0.297
25.	Deltapine			7.2	30.8	8.18	3.52	3.4	78	72	4.92	9.66	15.4	21.6	25.5	-0.214

* Acetylation Index-- $N = \frac{\log \frac{A}{T}}{\log T}$, where A = per cent acetyl at Time T.

NOTE: All samples whose sources were not listed were furnished by the Southern Regional Research Laboratory in New Orleans, La.

Final Report, Project No. 208-156

TABLE II

ACETYL VALUES FOR COTTONS SCOURED BY DIFFERENT METHODS

Cotton	Acetyl Values																				
	Unscoured			Scour I			Scour II			Scour III			Scour IV			Scour V			Scour VI		
	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)
Memphis	14.0	19.5	23.5	16.2	21.8	23.9	18.3	20.8	26.1	17.9	21.2	26.1	19.2	23.2	24.6	18.3	23.7	25.4	16.4	19.7	23.7
Empire (Bale 92)	8.00	13.5	18.8	14.2	19.4	22.7	16.3	20.9	24.2	16.4	20.6	25.3	17.0	22.9	23.7	16.7	22.7	24.5	15.1	20.0	21.5
Bob Shaw	5.95	10.6	15.6	14.1	19.0	21.8	16.1	19.7	23.8	14.9	20.0	24.8	16.0	21.5	23.0	16.3	21.5	24.2	13.9	19.0	20.9
Stoneville 2B (Bale 249290)	9.37	15.1	19.2	14.4	20.0	22.2	16.2	20.4	23.6	16.3	20.2	25.0	16.8	22.5	23.8	17.6	22.5	24.8	14.9	19.6	22.1
Acala 1517	7.95	14.0	20.3	15.0	20.6	22.9	15.9	21.1	25.0	17.9	21.5	25.0	17.0	22.7	24.6	18.0	23.3	26.0	15.8	20.6	22.4
Lockett 140	6.23	10.8	13.5	13.6	19.0	21.8	15.6	19.8	24.4	15.8	20.4	25.6	15.4	20.8	23.8	16.3	21.9	24.8	14.7	19.5	22.6

Methods:

I. Water at boil; 60 minutes.

II. 1.0% DuPonol RA, 1.5% $Na_4P_2O_7$; 30 minutes at the boil.

III. 2.0% NaOH, 1.0% Merpol C, 0.5% Duponol RA and 0.5% $Na_4P_2O_7$; 60 minutes at the boil.

IV. 3.0% NaOH, 0.1% Triton X-100 and 0.3% $Na_4P_2O_7$; 60 minutes at the boil.

V. 5.0% NaOH and 2.0% Neutral Oleate Soap; 90 minutes at the boil.

VI. 20% NaOH, 2.0% Igepon T and 4.0% N_3PO_4; 120 minutes at 255° F.

NOTE: Amounts of scouring ingredients are based on the weight of the cotton samples. Acetylation temperature was 64° F.

TABLE III

ACETYL VALUES FOR COTTONS CONDITIONED AT VARIOUS RELATIVE HUMIDITIES

Cotton	Per Cent Acetyl at 15% Relative Humidity			35% Relative Humidity			50% Relative Humidity			65% Relative Humidity			85% Relative Humidity		
	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)	30 (min)	60 (min)	90 (min)
Memphis	12.3	18.4	20.7	12.2	17.6	20.9	12.6	19.4	24.3	14.0	19.5	23.5	14.3	21.2	24.8
Empire (Bale 92)	5.70	10.6	15.9	5.39	9.67	15.3	6.31	11.7	17.5	8.00	13.5	18.8	8.26	14.7	18.6
Bob Shaw	3.87	7.59	12.2	3.57	6.89	12.6	4.47	8.82	14.6	5.95	10.6	15.6	5.76	11.3	15.6
Stoneville 2B (Bale 249290)	6.59	11.7	16.8	6.54	10.91	17.1	7.64	13.0	19.2	9.37	15.1	19.2	9.81	16.3	20.4
Acala 1517	6.51	11.3	17.0	6.90	11.8	17.3	8.25	14.0	19.7	7.95	14.0	20.3	11.4	19.1	22.9
Lockett 140	3.40	6.84	11.7	3.51	6.87	11.4	3.90	8.51	13.0	6.32	10.8	13.5	6.84	12.2	16.2

NOTE: Acetylation temperature was 64° F.

TABLE IV

ACETYL VALUES OF COTTON AFTER VARIOUS PRESOAKING TIMES AND TEMPERATURES

Cotton	Per Cent Acetyl at																				
	No Presoak	70° F Presoaking					100° F Presoaking					130° F Presoaking					170° F Presoaking				
		10 (min)	30 (min)	60 (min)	120 (min)	240 (min)	10 (min)	30 (min)	60 (min)	120 (min)	240 (min)	10 (min)	30 (min)	60 (min)	120 (min)	240 (min)	10 (min)	30 (min)	60 (min)	120 (min)	240 (min)
Memphis	3.72	8.07	10.5	13.1	14.1	14.3	16.9	15.5	17.2	18.1	18.5	17.3	17.1	17.5	19.2	18.6	16.5	17.0	16.6	16.8	16.6
Empire (Bale 92)	1.53	2.75	3.89	5.07	6.26	7.28	7.83	8.91	9.86	11.8	13.0	8.43	10.3	12.3	13.4	13.6	11.4	12.0	12.4	11.6	12.5
Bob Shaw	0.87	1.46	2.36	3.07	4.38	4.42	4.62	4.59	6.44	8.45	9.26	6.08	8.30	9.87	12.1	12.3	8.90	9.53	9.98	9.95	10.8
Stoneville 2B (Bale 249290)	1.37	3.37	3.97	4.87	6.80	8.03	7.90	8.23	11.5	13.8	14.8	11.6	12.6	13.9	14.6	14.7	12.8	13.0	13.3	12.5	13.9
Acala 1517	1.43	2.17	2.78	4.20	5.62	6.79	6.61	6.44	9.75	14.5	17.6	9.81	12.4	15.6	16.2	16.4	14.0	14.5	14.4	15.0	15.4
Lockett 140	1.02	1.37	1.84	2.59	2.62	4.04	4.33	4.74	5.45	9.06	10.6	6.21	7.42	9.91	11.1	10.8	8.86	9.27	9.81	9.75	11.4

NOTE: All cotton were acetylated for 45 minutes at 64° F.

TABLE V

THE EFFECT OF ACETYLATING TEMPERATURE ON ACETYL VALUES AND FIBER STRENGTH

Cotton	Acetylation Temperature 58° F Acetyl (%)	58° F Pressley Index	64° F Acetyl (%)	64° F Pressley Index	70° F Acetyl (%)	70° F Pressley Index	76° F Acetyl (%)	76° F Pressley Index	82° F Acetyl (%)	82° F Pressley Index
Memphis	12.7	6.02	16.5	6.89	19.2	6.09	20.7	5.52	23.7	--*
Empire (Bale 92)	9.13	6.62	11.4	6.91	13.5	6.49	17.1	6.16	20.4	--
Bob Shaw	7.48	7.70	8.90	6.68	10.7	6.84	15.3	6.33	19.3	--
Stoneville 2B (Bale 249290)	10.7	7.16	12.8	6.64	15.4	6.80	18.3	6.17	21.5	--
Acala 1517	10.8	8.01	14.0	8.29	16.8	6.93	20.0	6.83	23.1	--
Lockett 140	7.96	6.53	8.86	7.29	11.0	6.27	14.7	6.01	19.9	--

*Fibers too weak to give reliable strength figures.

Note: All samples were acetylated for 45 minutes.

for each cotton may be represented by the following equation:

$$\log \frac{A}{T} = \log C + N \log T$$

where: A = acetyl content in per cent

T = Time of acetylation in minutes

C = constant, and

N = constant, or "acetylation index".

From the above equation each cotton can be characterized by the value C or N.

The C value represents the acetyl value of the cotton when acetylation time is one minute. The N is the slope of the curve, $\log \frac{A}{T} = \log C + N \log T$. After several trial correlations of C and N with various physical properties, it was found that the N value best characterized the cottons. Therefore, the N value which is called "acetylation index," for each cotton was calculated by the method[3] of least squares and tabulated in Table I.

Simple correlations were made to determine if maturity, fineness, x-ray angle or other physical properties are related to the acetyl content of cotton represented by the acetylation index. In order to simplify the calculation, the six cottons used for scouring studies were selected as representative of the cottons available. Later the nine cottons for which wax determination were available were substituted so the relation between per cent wax and acetylation

[3] Arkin, Herbert, and Colton, Raymond R., An Outline of Statistical Methods, Fourth Edition, Barnes and Noble, New York, 1939.

could be calculated. It was also thought that if good correlations were obtained with a small number of cottons, probably much better correlations would result when the number of cottons correlated was increased.

Preliminary calculations on the six-cotton group gave significant results. With the excellent simple correlation on the six-cotton samples as a basis, multiple correlations using maturity by sodium hydroxide and arealometer methods, fiber fineness by micronaire and arealometer methods and per cent wax by the alcohol-chloroform extraction method were calculated for a nine-cotton sample. These results which are presented in Table VI show several significant correlations. The most significant correlation is acetylation index N vs. per cent wax and fiber fineness by the micronaire.

In general, it was noted that the per cent maturity by the arealometer method and the fiber fineness by the micronaire method gave the higher correlations as represented by the correlation coefficients. Since there were no wax values for additional cottons, the high correlation with per cent wax and micronaire fineness could not be verified with a larger number of cottons. However, maturity and fineness values were available for 14 additional cottons and multiple correlations were calculated for these 14 in addition to the original nine. The results of these correlations for the twenty-three cottons are shown in Table VI. These results show no significant correlations existing between the acetylation index and maturity plus fiber fineness.

A plot of acetylation index vs. maturity as well as acetylation index vs. fiber fineness reveals the reason fiber fineness and maturity were a good indication of the acetylation for the six and the nine cottons but no

indication for the twenty-three cottons. The N vs. maturity and N vs. fiber fineness plot for the nine cottons follows a fairly straight line but the additional points from the fourteen other cottons give a scatter diagram with no obvious trend. From this evidence it must be concluded that the significant simple correlations obtained with the six-cotton group and the significant multiple correlations obtained with the nine-cotton group are caused by a chance bias in selecting these cottons, i.e., these six and nine cottons are not representative of the twenty-three cottons used in the final analysis. It would not be proper, however, to exclude the possibility of significant correlation with the per cent wax with additional data, but in view of correlation results with maturity and fineness there is doubt that such correlation exists. It should be noted that Memphis cotton, which appears in all the studies because of its very low maturity, is a poor choice because there are no other cottons with similar properties and because it is not a variety of cotton. However, the Memphis cotton may indicate that in general an increase in the acetylation index may correspond with a low maturity. It should be reiterated that this is not a result that can be statistically verified with the present data but an observation which would require additional low maturity cottons (between 40 per cent and 75 per cent) for statistical analyses.

To summarize, the statistical studies show that the degree and rate of acetylation for different raw cottons cannot be predicted or determined with accuracy by their known physical and chemical properties.

B. Analysis of Variance

In order to determine the effect of the variables in the scouring experiments an analysis of variance was made using the data in Table II, Scours II through V. The results of this analysis are shown in Table VII A.

These results show that the largest effect is caused by time of acetylation. It also shows that while the effect of cotton variety is considerably less than the effect of acetylation time it is highly significant. The effects due to the interaction of the method of scouring with acetylation time and the variety of cotton with acetylation time also contribute to differences in acetyl content. The conclusion from examination of the raw data is that there is no significant difference in the acetyl values of the cottons acetylated after being scoured by any of the four scouring methods. This is confirmed by the statistical results. An examination of the results of the two-factor analysis (Table VIIB) shows the effect of acetylation time. At thirty minutes acetylation time there are no significant differences due to scouring methods while at sixty and ninety minutes the scours contribute significantly to the interaction of scour with time. The differences due to the varieties of cotton are highly significant for every level of acetylation time.

The residual mean square can be regarded as an estimate of the error in experimental process. The variation in the acetyl content for the scouring experiments is estimated from the mean square to be 0.45 per cent acetyl. This means that the acetyl value reported plus or minus two times 0.45 per cent acetyl or 0.90 per cent acetyl will include the true acetyl percentage approximately 95 per cent of the time.

A further comparison of the acetyl values from acetylation of scoured cottons with acetyl values from unscoured cottons shows that the acetyl contents are significantly greater.

The analysis of variance for the relative humidity conditioning studies shows that all the second-order interactions, i.e., per cent relative humidity

condition with cotton variety, cotton variety with acetylation time and acetylation time with per cent relative humidity condition, are highly significant. Therefore, in order to show the significance of the main effects of per cent relative humidity condition, cotton variety and acetylation time, three two-factor analyses were made (Table VIIIB). These results indicate that all the main effects are also highly significant. The acetylation time and cotton variety probably have more effect on acetyl content than the per cent relative humidity at which the cotton was conditioned. The estimate of error for the conditioning experiments is 0.44 per cent acetyl.

To sum up, the statistical analyses of cottons conditioned at various relative humidities prior to acetylation indicate that significant differences in acetyl content are caused by the moisture content of the cottons, the time of acetylation and variety of cotton as well as all the secondary effects which are the interactions between the main effects.

The analysis of variance for the presoaking experiments verify the conclusion reached by qualitative examination of the original data, i.e., the statistical results presented in Tables IXA and IXB show that the two main effects of presoaking time and cotton variety are highly significant. The effects of two second-order interactions cotton variety with presoaking temperature and presoaking temperature with presoaking time are also highly significant. The two-factor analysis of presoaking time and cotton variety for each presoaking temperature shows that while highly significant, the presoaking time has a decreasing effect with increase in presoaking temperature. The estimated error for the scouring experiments was 0.78 per cent acetyl.

TABLE VI

RESULTS OF CORRELATIONS BETWEEN THE PROPERTIES OF RAW COTTONS AND THE ACETYLATION* of THESE COTTONS

Number of Cottons Correlated	Properties Correlated	Correlation** Coefficient	Variance Ratio	Comment
9	N vs per cent wax + per cent Maturity (NaOH)	0.874	9.75	Correlation significant (between 5 and 1 per cent level)
8	N vs per cent wax + per cent Maturity (Aeralometer)	0.868	7.62	Correlation significant (between 5 and 1 per cent level)
9	N vs per cent + Fineness (Aeralometer)	0.863	8.76	Correlation significant (between 5 and 1 per cent level)
9	N vs per cent wax + Fineness (Micronaire)	0.927	18.2	Correlation highly significant (at 0.5 per cent level)
8	N vs per cent Maturity (Arealometer) + Fineness (Arealometer)	0.684	2.21	Correlation not significant (above 20 per cent level)
8	N vs per cent Maturity (Arealometer) + Fineness (Micronaire)	0.881	8.7	Correlation significant (between 5 and 1 per cent level)
9	N vs per cent Maturity (NaOH) + Fineness (Micronaire)	0.792	5.04	Correlation not significant (between 20 and 5 per cent level)
9	N vs per cent Maturity (NaOH) + Fineness (Arealometer)	0.646	2.14	Correlation not significant (at 20 per cent level)
23	Per cent Maturity (Arealometer) + Fineness (Arealometer)	0.321	1.09	Correlation not significant (above 20 per cent level)
23	Per cent Maturity (Arealometer) + Fineness (Micronaire)	0.210	0.41	Correlation not significant (much above 20 per cent level)

* Acetylation Index $= N = \frac{\log \frac{A}{T}}{\log T}$, where A = per cent acetyl at T = Time.

** In general a correlation coefficient of 1.0 indicates perfect correlation.

TABLE VIIA

ANALYSIS* OF VARIANCE** FOR SCOURED COTTONS

Source of Variance	Degrees of Freedom	Sum of Squares	Mean Squares	Comments
Method of Scour	3	1,205	402†	Since interactions of method of scour with time of acetylation and cotton variety with time of acetylation are significant, the main effect of time of acetylation is shown by the two-factor analysis of method of scour with variety of cotton for each acetylation time.
Cotton Variety	5	3,031	606†††	
Time of Acetylation	2	75,941	3,797†	
Interaction between: (1) Method of Scour and Time of Acetylation	6	1,754	292†††	
(2) Cotton variety and Time of Acetylation	10	468	47††	The estimate of error for the scouring experiments is ± 0.45 per cent acetyl.
Residual	45	884	20	
TOTAL	71			

*Three-factor analysis.

**For complete explanation of Analysis of Variance see: Brownlee, K. A., Industrial Experimentation, Fourth Edition, Her Majesty's Stationery Office, London, 1949.

†Not significant (at 5 per cent level or above).

††Significant (between 5 per cent and 1 per cent level).

†††Highly significant (above 1 per cent level).

TABLE VIIB

ANALYSIS* OF VARIANCE FOR SCOURED COTTONS

Source of Variance	Degrees of Freedom	Mean Squares: Acetylation Time at T_1	Mean Squares: Acetylation Time at T_2	Mean Squares: Acetylation Time at T_3
Method of Scour	3	77**	697***	213***
Variety of Cotton	5	383***	179***	138***
Residual	15	31	12	16
TOTAL	23			

*Two-factor analysis.

**Not significant (at 5 per cent level or above).

***Highly significant (above 1 per cent level).

TABLE VIIIA

ANALYSIS* OF VARIANCE FOR CONDITIONED COTTONS

Source of Variance	Degrees of Freedom	Sum of Squares	Mean Square	Remarks
Per Cent Relative Humidity Conditionings	4	2,169,036	542,249	Since all interactions are highly significant, the significance of the main effects can be shown by two-factor analyses.
Cotton Varieties	5	8,971,885	1,794,377	
Time of Acetylation	2	14,908,806	7,454,403	
Interaction between: Per Cent Relative Humidity and Cotton Variety	20	193,303	9,665**	
Cotton and Time of Acetylation	10	171,209	17,121**	
Time of Acetylation and Per Cent Relative Humidity	8	115,274	14,409**	The estimate of error = ± 0.44 per cent acetyl
Residual	40	77,979	1,949	
TOTAL	89			

*Three-factor analysis.

**Highly significant (above 1 per cent level).

TABLE VIIIB

ANALYSIS* OF VARIANCE FOR CONDITIONED COTTONS

Source of Variance	Degrees of Freedom	Mean Squares: Cotton 1	Cotton 2	Cotton 3	Cotton 4	Cotton 5	Cotton 6
Acetylation Time	2	1,217,180†	1,375,034**	1,105,992**	1,391,429**	1,570,476**	874,949**
Relative Humidity Condition	4	54,275**	79,910**	68,435†	93,298**	181,022**	113,652**
Residual	8	5,398	2,430	2,813	3,934	4,858	3,713

Source of Variance	Degrees of Freedom	Mean Squares: Relative Humidity at H_1	Relative Humidity at H_2	Relative Humidity at H_3	Relative Humidity at H_4	Relative Humidity at H_5
Acetylation Time	2	1,303,639**	1,334,065**	1,767,515**	1,468,520**	1,638,525**
Cotton Variety	5	370,801**	370,154**	413,730**	300,830**	377,518**
Residual	10	5,793	4,985	3,723	8,038	2,237

Source of Variance	Degrees of Freedom	Mean Squares: Acetylation Time at T_1	Acetylation Time at T_2	Acetylation Time at T_3
Relative Humidity Condition	4	110,883**	275,813**	187,538**
Cotton Variety	5	474,424**	721,512**	636,822**
Residual	20	3,221	5,504	3,805

*Two-factor analyses.

**Highly significant (above 1 per cent level).

†Significant (between 5 per cent and 1 per cent level).

TABLE IXA

ANALYSIS* OF VARIANCE FOR PRESOAKED COTTONS

Source of Variance	Degrees of Freedom	Sum of Squares	Mean Square	Remarks
Presoaking Temperature	3	9,815,717	3,305,143	Significance of the presoaking temperature effect is shown by the two-factor analysis of cotton variety and presoaking time for each presoaking temperature
Presoaking Time	4	2,161,164	543,607**	
Cotton Variety	5	9,843,617	1,989,686**	
Interaction between:				
Cotton Variety X Presoaking Temperature	15	630,690	42,046**	Estimate of error = ± 0.78 per cent acetyl
Presoak Time X Presoaking Temperature	12	686,214	57,185**	
Residual	80	481,678	6,021	
TOTAL	119			

*Three-factor analysis

**Highly significant (above 1 per cent level).

TABLE IXB

ANALYSIS* OF VARIANCE FOR PRESOAKED COTTONS

Source of Variance	Degrees of Freedom	Mean Squares: Presoak Temp. at T_1	Presoak Temp. at T_2	Presoak Temp. at T_3	Presoak Temp. at T_4
Presoaking Time	4	181,927**	339,400**	157,625**	7,890**
Cotton Variety	5	579,280**	634,187**	485,222**	398,170**
Residual	20	4,798	15,885	5,819	963

*Two-factor analysis.

**Highly significant (above 1 per cent level).

V. GENERAL CONCLUSIONS

The over-all conclusions which may be drawn from the data are summarized below. Attention is called to many graphical illustrations of data which are not included in this report but have been presented in quarterly progress reports.

This project has resulted in the development of equipment and techniques such that data obtained are sufficiently reproducible to permit statistical analyses.

1. Correlation studies of the data obtained in these studies show that the degree and rate of acetylation cannot be predicted with accuracy from the physical and chemical properties of the raw cottons used.

2. Scouring cotton prior to acetylation tends to minimize the differences in the acetylation reactivity of the different cottons.

3. Scouring increases the rate and degree of acetylation.

4. The variety of cotton, time of acetylation, the action of variety with acetylation time and the action of method of scouring with time of acetylation, all significantly influence the acetylation reaction.

5. High relative humidity conditioning prior to acetylation slightly increased the rate and degree of acetylation of the cottons studied.

6. A high presoaking temperature of glacial acetic acid increased the rate in which maximum acetylation is reached under constant acetylation conditions.

7. An increase in acetylation temperature increases the degree of acetylation which is also accompanied by an increase in degradation of the acetylated fiber.

8. Correlation data show that all factors, such as cotton variety, time of acetylation, relative humidity condition, relative humidity with acetylation time and relative humidity condition with cotton variety, influence the acetylation reaction. The relative humidity affects the acetyl values much less than either cotton variety or acetylation temperature.

VI. RECOMMENDATIONS

Although statistical analyses of the data obtained showed several significant correlations, the data were insufficient to permit the development of accurate formulas showing the interrelationship as desired in the scope of the contract proposal. It is, therefore, recommended that:

1. Additional studies be made on a single cotton variety having wide variations in maturity values. This would yield data which would permit correlations of physical and chemical properties with maturity.

2. The program be extended to include several varieties of cottons having widely different physical properties.

3. Make statistical analyses of all data from the previously mentioned studies to derive a basis for prediction of the acetylation reactivity of cotton.

Respectfully submitted:

James L. Taylor
Project Director

Alton R. Colcord, Jr.
Research Assistant

Approved:

Herschel H. Cudd, Director
Engineering Experiment Station